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DE-92148-TOP-01

DIRECT LIQUEFACTION PROOF-OF-CONCEPT FACILITY

Hydrocarbon Research, Inc., Lawrenceville, N.J.

FINAL

Technical Progress Report
POC Run 01 (260-04)

Work Performed Under Contract No. AC22-92PC92148

For
U.S. Department of Energy
Pittsburgh Energy Technology Center,
Hydrocarbon Research Inc., Princeton, NJ,

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SECTION I

ABSTRACT

This report presents the results of work conducted under the DOE Proof of Concept Program in direct coal liquefaction at Hydrocarbon Research, Inc. in Lawrenceville, New Jersey, from October 1992 through April 1994. The work included extensive modifications to HRI's existing 3 ton per day Process Development Unit (PDU) and completion of the first PDU run (POC Run 1) under the Program. The 58-day POC Run 1 demonstrated scale up of the Catalytic Two-Stage Liquefaction (CTSL Process) on Illinois No. 6 coal to produce distillate liquid products at a rate of up to 5 barrels per ton of moisture-ash-free coal.

During the first fiscal year, the major effort was to modify the PDU to improve reliability and to provide the flexibility to operate in several alternate modes. The Kerr McGee Rose-SR^{SM} unit from Wilsonville, Alabama, was redesigned and installed next to the U.S. Filter installation to allow a comparison of the two solids removal systems. Also included was a new enclosed reactor tower, upgraded computer controls and a data acquisition system, an alternate power supply, a newly refurbished reactor, an in-line hydrotreater, interstage sampling system, coal handling unit, a new ebullating pump, load cells and improved controls and remodeled preheaters.

The 58-day CTSL Illinois coal demonstration run achieved several milestones in the effort to further reduce the cost of liquid fuels from coal. This was the first demonstration of HRI's Catalytic Two Stage Liquefaction technology at the 3 ton per day scale and featured many improvements over the earlier testing conducted at the Wilsonville, Alabama Pilot Plant. Distillate liquid yields of 5 barrels per ton of moisture ash free coal (about 75 wt% on MAF coal) were achieved. Coal slurry recycle rates were reduced from the 2-2.5 to 1 ratio demonstrated at Wilsonville to as low as 0.9 to 1 during the recent test. This greatly improves the process efficiency, process performance and economics. Coal feed rates were increased during the test by 50% while maintaining process performance at a marginally higher reactor severity. This offers the potential for further reduction of commercial plant investment per unit of coal feed. Sulfur in the coal was reduced from 4 wt% to about 0.02 wt% sulfur in the clean distillate fuel product. More than 3500 gallons of distillate fuels were collected for evaluation and upgrading studies by DOE and their contractors. The ROSE-SR^{SM} Process was operated for the first time with a pentane solvent in a steady-state mode. The energy rejection of the ash concentrate was consistently below prior data, being as low as 12%, allowing improved liquid yields and recovery.

SECTION II

EXECUTIVE SUMMARY

This report is prepared under a multi-year Proof-of-Concept direct coal liquefaction program funded by the U.S. Department of Energy's Pittsburgh Energy Technology Center, Kerr-McGee Corporation, and Hydrocarbon Research, Inc. (HRI). The program is directed toward scaling up and demonstrating new liquefaction concepts that can potentially lower the cost of synthetic liquid fuels to less then $30 per barrel. The work reported herein includes modifications to the Proof-of-Concept facility at HRI's Lawrenceville, New Jersey, R&D Center and completion of a 58-day demonstration run on Illinois No.6 bituminous coal in a Catalytic Two-Stage Liquefaction mode. Operations at the 3 ton of coal per day facility produced yields of five barrels of clean distillate liquid products per ton of coal. The high quality liquid products can be readily refined into gasoline and diesel fuel.

The 58 day demonstration run processed a high sulfur (4 wt%) Illinois bituminous coal using HRI's Catalytic Two-Stage Liquefaction (CTSL) Technology. The process is similar to that utilized in HRI's commercially demonstrated H-Oil® Process for heavy oil conversion. In the CTSL Process (*See Figure 3.1*), the first stage reactor operates at lower temperature (385-415°C) to hydrogenate the coal and recycle oil, while the second stage operates at a higher temperature (425-440°C) to convert the coal and heavy oils to clean distillate liquid products. The products can be utilized for gasoline, jet fuel, or diesel transportation fuels, or as home heating utility or combustion turbine fuels. Unconverted coal and ash are separated from recycle oils and valuable products using solids separation techniques such as filtration or solvent extraction. Kerr-McGee's ROSE-SR^{SM} solids separation technology was demonstrated during the Illinois coal demonstration run.

The scale-up of the CTSL process in POC Run 01 on Illinois #6 Coal was the culmination of a ten year effort devoted to the development of this two stage ebullated-bed reactor system using a low to high temperature sequence.

POC-01, the first PDU Run of this program, was completed on February 19,1994, after 58 days of on-stream coal operations.

Some of the major accomplishments from the run were:

- Successfully commissioned and operated the newly installed equipment and the completely integrated two-stage coal liquefaction unit, including the ROSE-SR^{SM} solids-separation unit.

- Achieved operation with a more concentrated coal feed slurry at a 0.9 to 1.0 oil/coal ratio. This greatly improves the process efficiency and economics as compared to the 2-2.5 to 1.0 ratios demonstrated at Wilsonville.

- Successfully operated the ROSE-SR^{SM} unit using a pentane solvent in a steady-state mode. Demonstrated energy rejection of the ash concentrate consistently below prior data, achieving 12% energy rejection for a sustained period.

- Collected 3500 gallons of distillate product (IBP to 350°C) for upgrading studies and engine testing.

- Demonstrated distillate (C_4-524°C) production at MAF levels of 70-74% and coal conversions of 95-96% with Illinois #6 Crown II Mine bituminous coal. *(See Table 3.1 following this section.)*

- Produced an IBP-350°C product with an API gravity of 33, nitrogen content of 0.06 wt% and a sulfur level of 0.03 wt%.

- Identified several design improvements for the ROSE-SR^{SM} unit, Hot Separator and Coal Feeding System.

- Met and exceeded total distillate product yields achieved earlier at Wilsonville with Illinois No.6 coal in a Catalytic Two-Stage Liquefaction Mode.

- Collected samples from various process streams for other DOE contractors.

- Tested several materials of construction supplied by Oak Ridge Labs in the reactors and at elevated temperature locations downstream.

Several objectives were not achieved during this run and are being rescheduled for POC-2; they are:

- Operation of the in-line hydrotreater. After several days of operation by-passing around the fixed catalyst bed was indicated, and it was taken off-line.

- Operation of the U.S. Filter. By-Passing around the filter leaves was observed and confirmed later.

- Operation of the Interstage Sample System. Plugs occurred on the high pressure side of the sample tap. Only two interstage samples were obtained.

- Operation with true extinction recycle. With increasing asphaltene content of the bottoms stream in the latter stages of the run, true extinction recycle of the 360°C+ oils could not be sustained due to a decrease in the ROSE-SR^{SM} separation efficiency. Mixed solvents are planned to be used in the ROSE-SR^{SM} unit during future PDU operations.

Conclusions

The overall conclusions from the run based on observations and analytical results are:

- Within the limitations of the ROSE-SR^{SM} unit to recover resid, extinction recycle can be achieved.

- A clean, IBP-360°C (IBP-680°F) distillate (sulfur=450ppm and nitrogen=550ppm) can be produced without additional hydrotreating.

- The CTSL Process is operable at slurry oil/coal ratios as low as 0.9-1.0.

- The ROSE-SR^{SM} Process separation efficiency is highly dependent on the asphaltene content of the feed and the solvent utilized. Using pentane with a quinoline insolubles level of 33% in the feed, an energy rejection of 12.5% was achieved at a bottoms solids content of 65%.

- Crown II Mine Illinois #6 Coal is a good candidate for liquefaction with demonstrated coal conversions up to 96% and residual oil (524°C+) conversions of over 85%.

- Akzo AO-60 catalyst is a strong attrition resistant catalyst with high activity for coal liquefaction.

- The ROSE-SR^{SM} unit efficiency is unaffected by whether the liquefaction recycle system is operated with or without ashy recycle.

- Coal conversion, as measured in atmospheric bottoms product, and the ash concentrate indicate that retrograde reactions are not occurring in the ROSE-SR^{SM} unit as observed previously with higher boiling ROSE-SR^{SM} solvents.

Recommendations

- Operation of the ROSE-SR^{SM} unit must be improved to recover more of the asphaltenes for recycle and extinction. Use of a mixed solvent is recommended.

- The Inline Hydrotreater internals must be modified to prevent by-passing of the fixed catalyst bed.

- Further studies at higher coal feed rates (space velocities) are warranted to improve process economics.

- The reliability of the catalyst addition system needs to be improved.

- Other areas that require redesign for improved operability are:

  Oil/Water Separation, External Separation, Let-down Valves, Slurry Heat Exchange, the U.S. Filter, the ROSE-SR^{SM} Bottoms Removal and Heat Exchange, Coal Feed System and the Interstage Sampling System.

- A further operation on bituminous coal with in-line hydrotreating and improved solid separation and heavy oil recovery is recommended.

SECTION III

INTRODUCTION

As a part of the National Energy Strategy an Advanced Research Strategic Thrust is identified as Advanced Research for Coal-Derived Liquid Fuels and has a primary objective "To evaluate novel concepts and establish the technology base for producing high quality hydrocarbon-based transportation fuels from coal to cost in the range of $25-$30/barrel of Crude Oil Equivalent. The advanced research thrusts focus on achieving objectives that support adaption of new technology into commercial practice in 5-10 years with some application in the near term (up to 5 years) as well. The Proof-of-Concept Program is the initial scale-up for direct coal liquefaction and establishes the basis of design for commercialization and proves the process economics. Under the Proof of Concept Program HRI was chosen to operate a two-stage Process Development Unit for a period of 3 years followed by two optional years.

The Department of Energy and Electric Power Research Institute (DOE & EPRI) operated a facility in Wilsonville, Alabama for over 10 years processing coal in various modes with single and two-stage reactors using dispersed and supported catalyst. In 1992 the DOE decided to close the Wilsonville facility (6 tons/day) and chose the smaller (3 tons/day) Hydrocarbon Research PDU facility, a less costly, more flexible system that could be operated part time. In September of 1992 Hydrocarbon Research Inc. was awarded a 3 year contract to modify and operate the existing 3 ton/day unit on a cost shared basis with Kerr-McGee as a participant.

Research and development objectives include scale-up of advanced direct liquefaction technology involving two stage reactions, co-processing of crude oils with coal, studies of alternate processing modes, evaluation of materials and equipment, improving product quality and reducing product cost. By the use of strategic feedstocks, commercially available catalysts, prototype equipment and improved design techniques and materials of construction efforts have been and will be focused on improving process economics. The PDU produces hydrocarbon distillates and by-products in sufficient quantity to allow various research activities, such as, product fractionation, upgrading, engine testing, storage stability, small scale combustion testing, and refining into chemical feedstocks.

Modifications were made to the HRI PDU to improve reliability and to provide flexibility for operation in several alternate modes. Included, were upgraded computer controls for automation and an alternate power supply to provide additional back-up in case of incoming power failure. The Kerr McGee ROSE-SR^{SM} unit from Wilsonville was modified to be a single-stage unit using a pentane solvent and installed next to the U.S. Filter system to allow for a direct comparison of the two solid separation systems. A new reactor, hydrotreater, interstage sample system, a coal handling system to receive pulverized coal, a new ebullating pump, and improved instrumentation were installed over a period of about one year. A major part of this installation was a new reactor tower enclosing the high pressure, high temperature vessels and upgraded preheaters.

The PDU is a totally integrated two-reactor-stage coal and oil hydrogenation process development unit. It includes coal and oil handling systems, slurry mixing, high pressure pumping, preheating, reaction, product separations, atmospheric and vacuum fractionation, naphtha stabilization, bottoms separation, product storage, data acquisition/storage/reporting and computer control. The PDU has been used to develop and scale-up the H-Oil® Process, H-Coal Process, Coal/Oil Co-Processing and CTSL processes. For this operation the PDU was equipped to remove solids via the ROSE-SR^{SM} critical solvent process, vertical leaf pressure filtration, or via vacuum distillation.

Phase I of the Proof-of-Concept Program consists of four PDU Runs preceded by equipment modifications. This report documents the PDU Modifications and the results from POC Run 1, a 58 day on-stream coal operation processing Illinois #6 Crown II Mine Bituminous Coal in the Catalytic Two-Stage Liquefaction mode (CTSL). A major objective was to operate with extinction recycle of the 370°C$^+$ fraction using the ROSE-SR^{SM} process for Solid Separation. Thirty-five hundred gallons of 60°C (140°F) to 349°C (660°F) equilibrium product was collected for upgrading studies. Results from this scale-up of the Catalytic Two-Stage Liquefaction Process are reported and compared with prior Bench Scale and Wilsonville data.

1. Program Objectives

The following are the objectives of the Proof-of-Concept Direct Coal Liquefaction Program.

Develop direct coal liquefaction and associated transitional technologies which are capable of producing premium liquid fuels, which are economically competitive with petroleum and which can be produced in an environmentally acceptable manner.

Focus on further developing Two-Stage Liquefaction by utilizing geographically strategic feedstocks, commercially feasible catalysts, and prototype equipment. Include testing co-processing or alternate feedstocks and improved process configurations.

- Demonstrate the operation of a two-stage catalytic ebullated-bed reactor system with bituminous and sub-bituminous coals (or lignite) using commercially available supported catalysts having good physical strength and activity for comparison with a slurry reactor system using dispersed catalysts and for comparison to prior bench scale and Wilsonville PDU results.

- Demonstrate variant liquefaction schemes, especially coal/oil co-processing, utilizing appropriate feedstocks with the scope of development depending on preliminary technical and economic evaluations. Co-Processing may enable early commercialization of coal liquefaction due to more favorable economics.

- Demonstrate satisfactory operation with alternate feedstocks. (Selection of another Illinois No. 6 coal and a lignite for pilot-scale tests is necessary, as Burning Star #2 coal and Martin Lake lignite that were used in the past may not be readily available in the future.)

- Focus on scale-up of PDU data to a commercial size unit by establishing operating parameters such as coal space velocity, bed exotherms, hydrogen gas rates/consumption, and reactor geometry/hydrodynamics.

- Prioritize process development for low-cost feedstocks based on distillate production rate and coal reactivity.

- Demonstrate suitable low-rank coal liquefaction conditions for obtaining low heteroatom and hydrocarbon gas yields and high coal conversions while eliminating potential solids deposition in the process units/lines.

- Obtain high distillate yields having good quality under low-severity conditions on a unit reactor volume basis.

- Demonstrate the economic viability of well dispersed, highly active catalyst (disposable as well as recoverable) for slurry reactor applications in two-stage liquefaction.

- Demonstrate optimum supported catalyst replacement rates with respect to coal throughput under steady-state catalyst activity conditions. Elucidate catalyst pore structure effects on reactant conversion and hydrogenation. Evaluate improved catalyst utilization concepts (e.g., regeneration, cascading).

- Produce premium products by in-line hydrotreating of distillate.

- Demonstrate improved hydrogen utilization in two-stage liquefaction by removing heteroatoms using pretreatment/preconversion methods (proven at bench-scale), especially for low-rank coals ($CO+H_2O$ is a possible candidate).

- Define and demonstrate two-stage liquefaction product properties (e.g., end-point) for economic upgrading and refining to make specification-grade products.

- Perform process development with strategically important high- and low-rank coals. When appropriate, select readily available low-ash coals that have good reactivity.

- Facilitate process development by studying the interaction between the first and second stages by developing appropriate sampling and analytical methods (e.g., evaluate conversions at preheater outlet, interstage, etc.).

- Demonstrate efficient and economic solids separation methods for different ranks of coal. Evaluate vacuum bottoms for determining the merits of schemes involving fluid or delayed coking.

- Study the merits of integrating advanced coal cleaning methods (e.g., agglomeration, acid washing/coal beneficiation, etc.) with two-stage liquefaction.

- Improve overall process operability by selecting and monitoring advanced equipment and instrumentation that have improved tolerance for material degradation while handling slurries containing fine particulates, heavy resids, and corrosive streams under high severity conditions.

2. Proof-of-Concept Run 1 Objectives

The following are the objectives of the first PDU run under the Proof-of-Concept Program:

- **To Ascertain Equipment Operability.**

  Included as new installations were another reactor rebuilt from a salvaged high-pressure vessel, an in-line Hydrotreater, Remodeled Preheaters, a new Coal Handling and Storage System, a redesigned and newly installed Kerr-McGee ROSE-SR^{SM} Unit, a repaired U.S. Filter System, an expanded Computer Control & Data Acquisition System, an On-Line Sampling System, a larger hot separator, rebuilt Hydrogen Compressors, new Catalyst Addition Valves, and a new Flare System.

- **To Provide a Tie Point with Wilsonville Data (Run 257J and Other).**

  Wilsonville Run 257 used Illinois #6 Coal and a Two-Stage Close Coupled Ebullated-Bed Reactor System with full and half reactor operation in both a high/low and low/high temperature operation. Condition 257J was the best performance obtained in the run and was used as a basis of design for an economic study by Bechtel for DOE.

- **To test the Rose-SR process and Filtration for solid/liquid separation.**

  A redesigned Kerr McGee ROSE-SR^{SM}, Critical Solvent Deasher from Wilsonville was to be tested using pentane solvent. A U.S. Vertical Leaf Pressure Filter similar to that in use at the British Coal LSE Pilot Plant was also available for study.

- **To Obtain Data on Catalyst Consumption, Coal Slurry Concentration and Extinction Recycle.**

  During POC-01 several levels of catalyst addition were used, from 1 to 4.5 lbs./ton coal with sufficient time to approach to equilibrium operation included. Coal slurry concentration was also planned as an integral part of this operation, oil/solids ratios from 1.5 down to 0.9 were planned. Extinction of the 370°C+ (700°F+) liquid product was incorporated to maximize the production of the more desirable light distillate. To accomplish this at maximum yield requires nearly complete recovery of the heavy oils from the solids containing bottoms, thus success of this objective is dependent upon efficient operation of the ROSE-SR^{SM} and/or Filter.

- **To Obtain Data on In-Line Hydrotreating.**

  Based on favorable Bench-Scale data, a Hydrotreater was designed and installed on the PDU to refine the hot separator light hydrocarbon overhead stream. The objective was to study the effect of time and temperature on hydrotreater performance and to demonstrate low heteroatom content of the distillate product.

- **To Collect Products for Evaluation and for other DOE Programs.**

  The DOE sponsored an upgrading study by Bechtel and others to produce transportation fuels from the distillate products of a coal liquefaction facility. A goal of collecting 2500 gallons of naphtha and distillate produced from extinction recycle operation was set. Other samples of distillate, heavy ends and bottoms products were also scheduled for collection for other DOE Programs.

- **To Evaluate Materials of Construction.**

  The Oak Ridge National Laboratory and the Japanese New Energy Development Organization (NEDO) supplied samples of various materials for study when exposed to coal liquefaction environments. Installations were in the reactors, separators and fractionators.

- **To Obtain Data for Commercial Design and Technical Assessment.**

  During the run and prior to shutdown, extensive data were collected including process yields, product qualities, stream properties, equipment performance, catalyst properties, solids-liquid separation performance, and effects of process operating conditions. These data form the basis for future commercial plant design and technical assessment.

TABLE 3.1
POC-01 PROCESS PERFORMANCE

Coal: Illinois No. 6 Crown II Mine (10.4 wt% Dry Ash)
Catalyst: Akzo AO-60 1/16" NiMo Extrudates in both Reactors

CONDITION	2
Process	CTSL
Period/s	24-26
Solids-Separation	ROSE-SR
Recycle Type	Ash-free
Coal Space Velocity, $Kg/hr/m^3$ (Stage)	310
$Lb/hr/ft^3$	19.3
K-1: Temperature, °C (°F)	407 (765)
Cat Replace. Rate, Kg/Kg Ton MF Coal	0.75
K-2: Temperature, °C (°F)	432 (810)
Cat Replace. Rate, Kg/Kg Ton MF Coal	1.50
Flow Rates	
Coal Feed, Kg/hr	70
Solvent/Coal Ratio, Kg/Kg	1.26
Material Balances	
Liquefaction Section Recovery, wt%	99.1
Overall Material Recovery, wt%	98.1
YIELDS, Wt% MAF COAL (Based on Liquefaction Section)	
H2S	2.45
NH3	1.45
H2O	9.91
COx	0.05
C1-C3	5.66
C4-177 °C (C4-350 °F)	19.03
177-288 °C (350-550 °F)	29.04
288-343 °C (550-650 °F)	17.52
343-524 °C (650-975 °F)	8.61
524 °C+ (975 °F+)	8.45
Unconverted Coal	4.97
Hydrogen Consumption	7.14

TABLE 3.1 (cont'd)
POC-01 PROCESS PERFORMANCE

Coal: Illinois No. 6 Crown II Mine (10.4 w% Dry Ash)
Catalyst: Akzo AO-60 1/16" NiMo Extrudates in both Reactors

PROCESS PERFORMANCE, Wt% MAF COAL

Coal Conversion	95
524 C+ Conversion	86.6
Desulfurization (Organic), Wt%	97.7
Denitrogenation, Wt%	82.5
C_4-343°C Net Distillates	65.6
C_4-524°C Distillates	74.2
, Barrels/MAF Ton	5.0
C_1-C_3 Selectivity, Kg/Kg of C_4-524°C (X 100)	7.6
H_2 Efficiency,Kg C4-524°C/Kg H_2	10.4

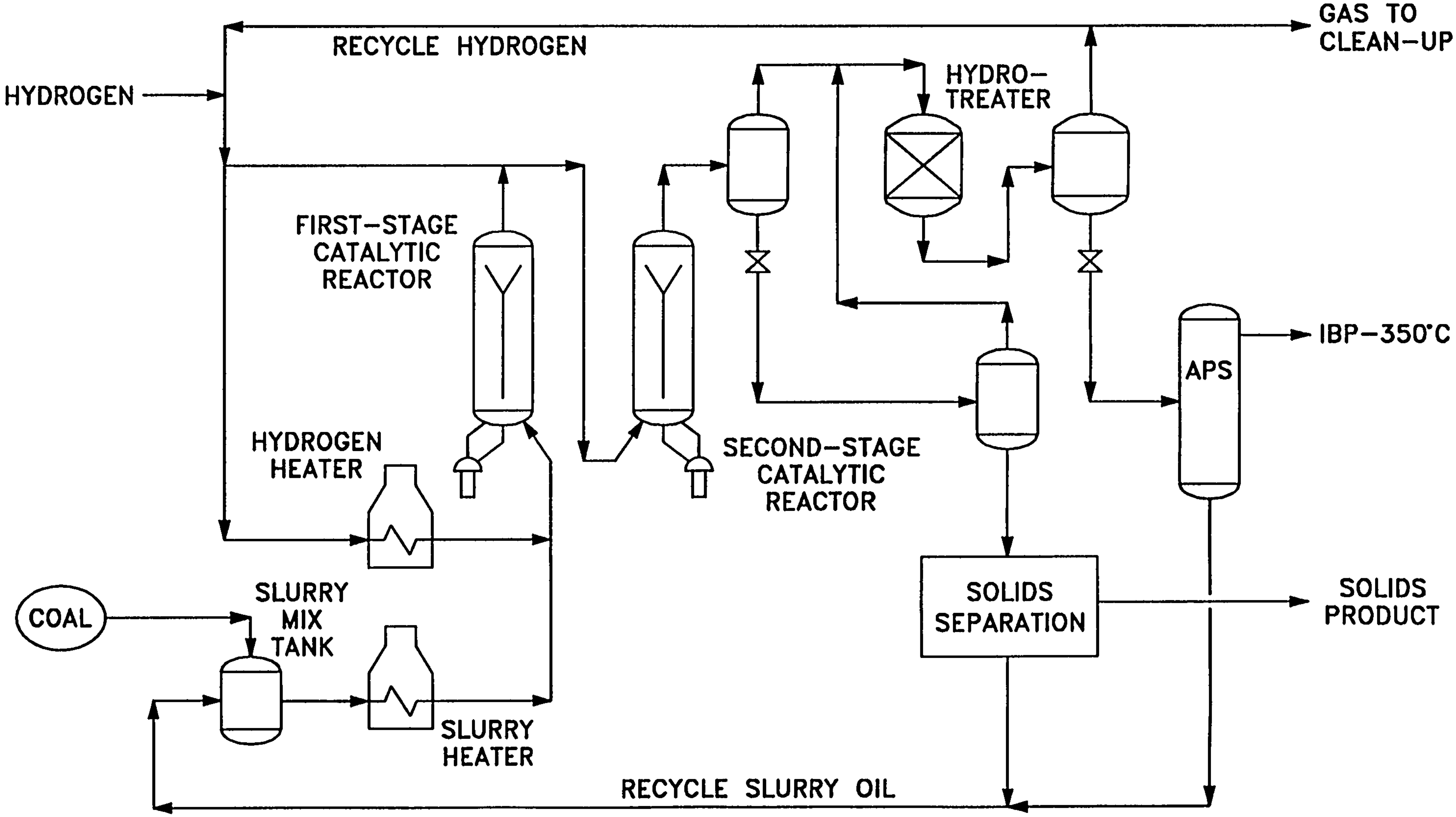
DEASHER PERFORMANCE

Organic Rejection, Wt% MAF	15.2
Energy Rejection, %	16.5
Deasher Coal Conversion, Wt% MAF	95.1

FIGURE 3.1

PROOF-OF-CONCEPT DIRECT LIQUEFACTION UNIT AT HYDROCARBON RESEARCH, INC.

SIMPLIFIED FLOW PLAN



SECTION IV

FEEDSTOCKS AND OPERATING SUMMARY

A. PROCESS DESCRIPTION OF THE PROOF-OF-CONCEPT RUN POC-01

The first Run in the Proof-of-Concept Program was carried out in HRI's Process Development Unit, Unit 260, which consists of two close-coupled ebullated bed reactors in series to convert coal and/or residual oil to high quality distillate fuels (*Figure 4.1*). The PDU is capable of operating at temperatures of up to 465°C (865°F) and pressures up to 20.7 MPa (3000 psig). Feed capacity can be as high 4 tonnes of coal per day. HRI's PDU is a totally integrated coal liquefaction/oil hydrogenation process development unit that includes coal and oil feed handling systems, slurry mixing (P-4), pumping (J-1), preheating sections (L-1 and L-2) besides two close-coupled ebullated bed reactors, product separators, atmospheric and vacuum fractionators, naphtha stabilizer, bottoms handling/recovery units, product storage, and on-line data acquisition (with computer control)/storage/reporting facilities. The HRI PDU facilities have been used in the past for the development of the H-Oil and the H-Coal Processes, for Coal-Oil Coprocessing Operations, and for Jet Fuel Production from shale oil for the U.S. Air Force.

One of the main objectives of the PDU Run POC-01 was to demonstrate HRI's CTSL Technology with extinction recycle operations (recycle of all 400°C+ heavy oil) while processing a high volatile bituminous Illinois No. 6 coal and show the effectiveness of the in-line hydrotreating which takes advantage of the reactor systems' severity. For the demonstration of the CTSL Technology as a part of the Proof-of-Concept Program, during Run POC-01, the Unit was configured with ebullating pumps for both reactor stages, the catalyst addition/withdrawal systems, and an in-line fixed bed hydrotreater. Three different solid separation options were executed during this run to demonstrate their steady-state on-line operability and their respective performance in terms of rejection of the organic material and energy associated with it. The lower these two numbers, the better is the solid separation scheme. The solid separations schemes used during POC-01 are discussed later as a part of the Unit Flow Scheme.

Before the startup, both reactors (K-1 and K-2) were charged with predetermined amount of the Akzo AO-60 catalyst extrudates, which was then presulfided using TNPS (Tri-Nonyl Poly-Sulfide) while being ebullated in gas oil. During the continuous operations, the feed mixture, consisting of coal (from a hopper, charging coal to the slurry mix tank, P-4, under pressure using a screw-feeder), recycle slurry oil, and hydrogen is pressurized and preheated prior to being introduced into the reactor. Recycle gas (about 80-85% hydrogen), pressurized and preheated, is also introduced into the reactor. This mixture then enters the first ebullated catalyst bed; the effluent from first reactor stage flows directly to the second stage reactor. An on-line sample withdrawal system installed on the ebullating line (internal recycle line) of reactor stage one can be activated to collect the interstage slurry samples for characterizing the performance of the individual reactor stages. Additional recycle gas is charged to the second stage reactor to control the inlet slurry temperature and to maintain an adequate hydrogen partial pressure. Second-stage reactor effluent, consisting of various liquid and gaseous products, unconverted feed and unreacted hydrogen, enters into a hot separator (O-1). The operating conditions for reactor stages and different separators are addressed in later sections.

The overhead effluent from the hot separator, consisting primarily of unreacted hydrogen, gaseous reaction products, and lighter distillates, passes through an in-line hydrotreater (K-3) that employed fixed-beds (baskets) of Criterion 411 catalyst with an intermediate quenching zone. Hydrotreater effluent is mixed with water to prevent plugging due to (ammonium chloride/ammonium sulfide) salt formation and cooled in a heat exchanger (M-2) before entering another flash vessel (Cold Separator, O-5). This vessel separates the hydrogen and gaseous products from the lighter liquid distillates. The overhead gases are scrubbed with No. 2 fuel oil (N-1), and the resulting hydrogen-rich gas (mol. wt. between 3-5) is pressurized and recycled to the reactor. The liquid flash bottoms are depressurized and sent to an oil-water separator (O-45).

O-1 separator bottoms, containing the heavier slurry product are depressurized and undergone a flash separation in the reactor liquid flash vessel, O-13. The resultant bottoms product from this vessel is partly sent to the recycle oil tank (O-43) in the ashy-recycle mode of operation. The rest of the bottoms slurry goes to a solids separation section (details to follow) which removes solids and recovers oil for recycle. In the ash-free recycle mode of operation, the entire O-13 bottoms go to the solids separation section for the recovery of the solids-free recycle oil stream.

The vapors from O-13 are cooled and flashed in vessel O-12 with the condensate entering the atmospheric still (N-2). The other feed stream to the atmospheric still is the cold separator O-5 bottoms (after passing through an oil-water separator, O-45). Atmospheric still bottoms are primarily sent back to the recycle tank O-43 to generate enough slurrying oil for the desired solvent-to-coal ratio. Any atmospheric still bottoms produced in excess of that needed for recycle is stored as a net process product. The atmospheric still overheads pass through a naphtha stabilizer (N-5) which removes impurities such as hydrogen sulfide and light hydrocarbons. Liquid from the stabilizer column is collected as naphtha product from the process. All noncondensables from the unit are flared.

PDU 260, during Run POC-01, had the flexibility to be operated in three different solids separation modes:

- Vacuum Distillation Mode
- ROSE-SR^{SM} Mode
- Filtration (vertical leaf U.S. Filter) Mode

In general, as described earlier, O-13 flash vessel bottoms product is sent to the O-13 liquid surge drum, O-46. From this vessel the solids containing stream is sent to one of the three solids separation options.

Vacuum Still Mode (*Figure 4.2*): For the vacuum still option, a portion of the material from O-46 is sent through the recycle holding drum, O-41, and the recycle weigh drum, O-43, to the slurry mix tank, P-4. The remainder of the material from O-46 is sent to the vacuum still feed accumulator, O-50, and then to the vacuum still, N-3. The solids containing vacuum still bottoms stream goes to storage, while the vacuum still overhead material goes to the flush/purge oil storage drum, O-42, and any excess overheads go to the clean oil tank, P-3.

ROSE-SR^{SM} Mode (*Figure 4.3*): For this route of solid separation, the material from O-13 bottoms goes to the vacuum still, N-3. The overheads from the vacuum still go to the flush/purge oil storage drum, O-42 and then to either purge oil day tank, O-40, or to the clean oil tank, P-3. The vacuum still bottoms stream goes to the ROSE-SR^{SM} section where the solids concentrated stream is sent to storage after separation and the solids-free oil goes to the recycle oil receiver (deasphalted oil).

Filtration Mode (*Figure 4.4*): During this mode of solid separation, the material from O-46 bottoms is sent to the filter feed drum, O-47, and then to filter, XF-1. From the filter, the solids concentrated stream is sent to storage. The solids-free stream is sent through the filtrate receiver, O-48, and the vacuum still feed accumulator, O-50, to the vacuum still, N-3.

B. FEEDSTOCKS

B.1 Feed Coal

The first PDU Run in the Proof-of-Concept was a pilot-scale demonstration of HRI's CTSL Technology carried out on a high volatile bituminous Illinois Seam No. 6 coal. The selection of a candidate coal for the PDU run was based on several criteria, mainly concerning coal-reactivity. Chemical and petrographic analyses of three Illinois No. 6 coals that were considered for the Wilsonville coal liquefaction program are shown in *Tables 4.1* and *4.2.* The following were the specifications on feed coal for PDU Run POC-01:

- Moisture Content: 7-9 Wt%
- Ash Content (Dry): ≤ 9W%
- Chlorine Content: < 0.20W%
- Particle Size: < 1W% on 50 mesh screen and
< 35W% through 350 mesh screen
- Drying & Grinding: Atmosphere containing < 3W% of Oxygen

The comparison of the Burning Star Mine No. 4 (strip mine, Perry County) coal with Illinois No. 6 from the Burning Star Mine No. 2 indicates that the Burning Star Mine No. 4 coal has similar volatile matter, slightly less ash, lower chlorine content, and higher total and pyritic sulfur content than Burning Star Mine No.2. The petrographic data indicate that the No. 4 Mine coal has marginally more reactive macerals than the No. 2 Mine coal. The conversions and distillate yields of these two Illinois No. 6 coals are expected to be similar (based on the reactivity projections using correlations developed by Gulf Oil for the SRC-II Process).

Similar comparison between the Crown II Mine (underground mine, Macoupin County) coal and Burning Star Mine Coals shows a slightly lower ash content and a higher volatile matter for the former coal. These coals have similar sulfur levels. The Crown II Mine coal has a higher reactive maceral content of 96.3% which is 2.3 % higher than the Burning Star Mine No. 2 coal. Based on these comparisons and analyses of coals provided by the Consol, Inc., small samples (one pound each) of Illinois No. 6 coals from the Burning Star Mine No. 4 and the Crown II Mine were acquired for an initial laboratory testing/screening program using microautoclave coal evaluation in support of PDU Run POC-01 (see Laboratory Support Section).

A small sample of Illinois No. 6 coal mined from the Burning Star Mine No. 4 was received from Consolidation Coal Co., while a sample of the Crown II Mine coal was received from Freeman United Coal Mining Co. After receiving these candidate coal samples, a series of microautoclave tests was to determine their reactivity and suitability for the PDU Run. The two coal samples had similar coal and resid reactivities. The Burning Star Mine No. 4 coal resulted in 1-2 Wt% higher THF conversion; however, the corresponding 524°C+ resid conversion was 1-2 Wt% lower than that of the Crown II Mine coal. Unfortunately, at the time the order for the coal was placed the Burning Star coal was not available due to labor problems at the mine. As a result, the Crown II coal was selected as a substitute for PDU Run POC-01.

B.2 Coal Preparation

Feed Coal Grinding Operations

The Illinois No. 6 coal from the Crown II Mine from United Freeman Coal Mining was ground under specified conditions by Empire Coke Company in Alabama.

HRI's specifications for the coal grinding were a moisture content of 2 to 5 wt%, 99 wt% passing thru 50 mesh, less than 35 wt% passing thru 325 mesh, and to control oxidation. During actual grinding operations, the control of the moisture level in the coal was adequate. Grinding operations were carried out under nitrogen to control coal oxidation. As shown in *Tables 4.3 and 4.4,* all ground coals were smaller than 50 mesh. Also, with the exception of one sample, most samples contained less than 35 Wt% of less than 325 mesh.

Table 4.5 lists the results obtained on two grab samples of coal, one of the raw coal and one of the ground coal, taken by HRI at Empire Coke Co. The Empire Coke Analyses of the ground coal batches can be found in *Table 4.6.* Also presented here is the important information from the operator logsheets from the grinding operation at Empire Coke Co. As seen from *Table 4.5*, both the grab samples contained very small amounts of sulfate sulfur (0.01 Wt% dry basis), indicating minimum surface oxidation of coals during grinding and handling operations.

B.3 Start-up/Make-up Oil

The Startup operations for the PDU involve preheating, catalyst presulfiding, and initial catalyst bed ebullation. All these steps require a continuous passage of an oil of appropriate physical and chemical properties through the unit. The oil is also needed for slurrying the initial batch of the feed coal. Any emergency unit shutdowns also need oil for flushing. This oil, called the start-up oil, can also be used later during the progress of the run as a make-up oil that is mixed with the process-generated recycle solvent to achieve the desired solvent-to-coal ratio. The plans for preparation of an appropriate start-up or make-up solvent included: using HRI's in-house solvent, L-769 (Tank No. 4 material) for blend (after topping off 343°C$^-$ material) with a decanted slurry oil (343°C$^+$), derived from catalytic cracking operations (purchased from an oil company). The start-up oil, so derived, was to be hydrotreated during the PDU start-up to improve its solvent-quality (as this hydrotreated oil is used as a make-up oil later in the run, whenever needed).

B.4 Startup Solvent Screening/Tests

A number of different solvents, especially petroleum-derived decanted oils and some of HRI's in-house solvents were tested for solvent-quality for coal liquefaction by using the standard "Equilibrium Solvent Qualification Tests" in microautoclaves (see the section on Laboratory Support). Following are the solvents/oils tested either as candidates for the PDU start-up or for establishing a base-line for comparison.

Solvents/Oils Screened for PDU Start-up	
HRI-Solvent Number	Description of Solvent
Topped L-769	A blend (from Tank No. 4) of Wilsonville distillate and oil recycle oil derived from PDU-003 sub-bituminous coal operations; 343°C$^-$ topped off.
HRI-5669	Petroleum-based light cycle oil (stored in Tank No. 5)
HRI-5737	Petroleum-based oil-Tonen heavy coker gas oil
HRI-5667	Petroleum-based vacuum gas oil
HRI-6172	Cat. Cycle Oil received from the Mobil Oil Co. (Joliet, Illinois)
HRI-5198	Standard HRI solvent for "Qualification Testing", obtained from Wilsonville coal operations
L-799	A blend of L-769 (Tank No. 4) with HRI-5669 (Tank No. 5)

Solvent Preparation

HRI already had about 5000 gallons of L-799 in Tank No. 5 (a blend of L-769 and HRI-5669). After acquiring about 6000 gallons of the FCC cycle oil from Mobil's Joliet Refinery, the oils were blended and stored in Tank No. 5 (the new blend was labeled L-800). This blend, L-800, was used as a start-up oil for the PDU. It was also analyzed in detail. Its properties, along with some other candidate solvents, are shown in *Table 4.7*. During the start-up operations, L-800, the Tank No. 5 material, was hydrotreated in the PDU while the reactor temperatures were still lower than about 700°F so that the conditions were favorable for hydrogenation, especially aromatic ring saturation reactions. The vacuum still was operated to cut oil at 343°C, and the vacuum still bottoms were collected in Tank No. 4. The hydrotreated oil was designated as L-803. This material was also analyzed in detail to determine the effect of hydrotreating. As shown in *Tables 4.7* and *4.8*, there is improvement is the H/C ratio of the oil after hydrotreatment besides improvement in its donatable hydrogen content (which is proportional to the weight percent of the cyclic protons, as estimated from ^{1}H-NMR). Significant sulfur and nitrogen removal was also achieved during hydrotreatment. Thus, the hydrotreated material, L-803, should be a reasonably good solvent for coal liquefaction. *Figure 4.5* shows the schematic of the solvent preparation.

C. CATALYSTS FOR POC-01

The POC-01 PDU Run was carried out with two ebullated catalyst bed reactor stages. The catalyst used in both the stages was a supported Ni-Mo on alumina catalyst, manufactured by Akzo (AO-60). This catalyst was in the form of 1/16" size extrudates and was tested earlier at HRI in one of the bench scale runs during the CTSL Project (Run CC-16). As shown in *Table 4.9*, which lists the physicochemical properties of the AO-60 catalyst, the Akzo catalyst compares very favorably with Shell S-317 catalyst (supported Ni-Mo 1/32" alumina extrudates) which was used by HRI earlier in most of the bench scale operations. Due to commercial unavailability of the Shell S-317 catalyst, the Akzo AO-60 catalyst was selected. At the beginning of the run, both reactor stages were charged with the desired initial inventories of the catalyst. The catalyst was presulfided using TNPS as a sulfiding agent during the startup operations.

The hydrotreater unit, K-3, that consisted of two catalyst beds housed in baskets separated by an inert containing section, was charged with a trilobe-shaped Criterion C-411 hydrotreating catalyst.

D. UNIT OPERATIONS

D.1 Run Plan

The actual run plan is shown in *Table 4.10* which details the operating conditions for the various conditions. A complete description of the conditions and operation of the unit is given in the Operations Summary section.

The primary objective of the first Proof of Concept operation was to demonstrate the CTSL process with Illinois No. 6 coal. Other objectives of the first Proof of Concept operation included:

- confirming the operability of the modified unit,
- evaluating ROSE-SR^{sm} solid separation technology,
- obtaining process performance data at different operating conditions regarding:
  - conversion and yields,
  - catalyst consumption,
  - extinction recycle and
  - in-line hydrotreating,
- generating product for subsequent detailed characterization.

Efforts were expended to achieve each of these objectives during this operation. The run was slated to be about sixty days long, with at least five different Process Conditions to investigate at the POC scale of operations. The selection of reactor temperatures and the low-high mode of operation was made based upon our earlier successful experiences under similar temperature sequencing at the bench-scale of operations at HRI. The ashy recycle mode was adopted in some conditions to complete the conversion of reactive macerals in feed coal and also to maintain an adequate concentration of the 524°C+ residuum in the recycle stream. To minimize the rejection of organics with the product solids, the ROSE-SR^{SM} was used as a back-end solids-separation unit during some conditions. The target catalyst replacement rate was 0.7 kg/T of feed coal for reactor K-1 and 1.4 kg/T for reactor K-2; this was decided based upon the values used in the economical assessments and commercial projections for coal liquefaction demonstration, both at HRI and elsewhere. This target replacement rate was approached starting with a half rate to expedite the deactivation or approach of the catalyst activity to a steady-state or an equilibrium-level. The hydrotreater, with a fixed-bed catalyst, was planned to be in-line throughout the run although this was not achieved due to the operational problems, elaborated in the later sections. The coal space velocity was increased from 320 to 480 kg/hr/m^3 of each reactor during the last three Run Conditions; the reactor temperatures were also raised during this time to provide similar overall process severity (STTUs). The recycle solvent to coal ratio of about 1.2 to 1.5 was employed during the run and towards the end, even a ratio of 0.9 was studied, as lowering this ratio was found to positively impact the process performance in one of our earlier bench operations with similar coal feed. Each Run Condition was allowed at least 6 to 7 days for achieving a steady-state so that more representative process data could be obtained for each of the Run Conditions. This had to be done as the inventories in the overall system were high and needed about 2 days for a complete replenishment.

Several modifications were also made to the PDU in order to get it ready for the POC-01 Run. In general, the new and modified equipment performed well. Additional equipment and procedural modifications have been identified to further improve unit operation (refer to Sections IV and V of this report for additional details).

Several accomplishments and highlights were achieved during this program including:

- demonstrated the CTSL Process with Illinois No. 6 coal feed
- processed 102 metric tons (112 tons) of as-received coal
- operated for 58 days on coal feed
- operated the ROSE-SR^{SM} section off-line
- operated the ROSE-SR^{SM} section to provide recycle oil
- hydrotreated 22,700 liters (6,000 gallons) of start-up oil prior to the run
- completed 4 different operating conditions
- obtained first stage reactor liquid samples
- obtained sour water samples to evaluate commercial waste water requirements
- mechanically tested the alternate hydrotreater feed system
- mechanically tested the hydrotreater
- tested the filter to generate recycle solvent
- obtained special product samples for outside testing and processing at:
    - Southwest Research Institute
    - PETC
    - UOP
    - Center for Advanced Energy Research
    - Ceramem
    - Alberta Research Council
- exposed corrosion coupons (provided by Oak Ridge National Laboratory) to the process. These coupons were in:
    - the first stage reactor
    - the second stage reactor
    - the hydrotreater
    - the top of the atmospheric still
    - the bottom of the atmospheric still
    - the vacuum still
- completed an environmental air monitoring program

There were eight coal outages in this run, including six times when all flows were stopped to institute mechanical repairs. Each of these outages was related to a mechanical, instrument or procedural item. (Refer to Section IV for details regarding the coal outages.) The causes of these have either been or will be addressed before the next PDU operation.

D.2 Major Unit Modifications

Major modifications were made to the 260 unit prior to the first Proof of Concept operation, POC-01 (Run 260-04). These modifications included:

- Installation of a ROSE-SR^{SM} system
- Installation of an in-line hydrotreater
- Upgrading the reactor tower and installation of a new ebullated bed reactor.
- Installation of a pneumatic coal handling system
- Installation of an alternate electrical backup to the facility
- Upgrading the data acquisition system to process control
- Rebuilding the make-up hydrogen compressors
- Replacing the catalyst addition/withdrawal valves
- Installation of a first stage reactor liquid sampling system
- Construction a spare ebullating pump
- Upgrading the preheater firing control system
- Relocating and upgrading the unit flare.

These modifications were performed between November 1992 and October 1993.

D.3 Operation Performance

The operation performance for the run is summarized in *Figures 4.6-4.11, 4.13* and *Tables 4.11 and 4.12.*

The material balance for this run was calculated two different methods, as an overall balance around the entire process and as a liquefaction balance which is only up to the Reactor Liquid Flash Vessel (RLFV). The latter method allows the performance of the liquefaction section to be separated from the performance of the solid separation system. The liquefaction material balance is also the mass balance that is later used to calculate all the normalized yields and process performance. These two methods of calculating the mass balance are presented in *Figure 4.6 and 4.7. Figure 4.6* shows the overall material balance for the run. As can be seen from the figure, each time the Unit experienced a shutdown the mass balance recovery percentage was greatly reduced. This is primarily due to the effect of inventory changes in the various holding vessels associated with the different solid separation systems. For the longest time of smooth operation, Period 11-32, the overall balance is close to 100 wt%. *Figure 4.7* shows the liquefaction section material balance recovery for the run. The average recovery for the entire run was 97.7 wt%. This probably averaged slightly below 100 wt% due to the difficulty in estimating the bottoms from the RLFV. This vessel empties into a holding tank (O-46) which was operated with a level transmitter and not a weigh scale. Converting a level to a weight always introduces some error. This has been modified to operate on a weigh scale for all future runs.

Figure 4.8 shows the average temperature for each reactor and how evenly they were held, outside of the line out periods. *Figure 4.9* shows the space velocity (based on coal) for the run. After each shutdown, during the line out periods, the coal rate was gradually brought up to the target rate as can be seen from this figure. During operations the space velocity varied from 320 kg/hr/m^3 (20 lb/hr/ft^3) to 480 kg/hr/m^3 (30 lb/hr/ft^3). The solvent to coal ratio (in the slurry mix tank) is presented in *Figure 4.10.* As can be seen from the figure, after each shutdown the coal concentration took about 2 days to build up to the desired level. *Figure 4.11* shows a material balance performed around the solid separation equipment. The two units used for this run were the ROSE-SR^{SM} and the Vacuum Still. The ROSE-SR^{SM} shows an excellent average balance of very close to 100 wt% over the course of the entire run. The vacuum still generally shows a good material balance, except near the startup periods. This was because the vacuum still is the solid separation system that the 260 unit is brought up on during startup, and it is during the startup that the material balance is the poorest. *Tables 4.11 and 4.12* show an end of run summary of the overall unit and the liquefaction section balances for all periods. The vessel inventory changes shown in these two tables can have a larger value than the actual capacity of the pertinent vessel because during the different shutdowns some of the vessels could have been emptied or filled.

D.4 Operations History

A summary of the operating history is presented in *Table 4.13a - 4.13e* and in *Figure 4.12.*

Startup Preparations

After the modifications were completed, the unit and the corresponding drawings were reviewed to confirm conformance. The unit was insulated, loaded with catalyst and pressure checked. Operating procedures were written, reviewed and issued.

hydrotreater. The initial loading of 45 kg (100 lbs) of Akzo AO-60 nickel-molybdenum catalyst (HRI-6043) was installed October 15, 1993, into each of the ebullated bed reactors. The remaining 11 kg (25 lbs) of catalyst needed to achieve the ebullated bed design load was added after start-up.

Approximately 22,700 kg (6,000 gallons) of Mobil cat-cycle oil arrived October 17, 1993. This was blended with the L-799 in Tank 5 to make a single start-up oil, L-800 for the run. This start-up oil was then hydrotreated and the 343°C- (650°F-) oil distilled out prior to coal feed.

Start-Up

Oil flows to the unit were started October 21, 1993, with the first and second stage reactors ebullated the next day. The unit was then lined out with the reactors at about 343°C (650°F) and 18.6 MPa (2700 psig) in order to hydrotreat and distill 227 kg/hr (500 pph) of start-up oil until October 28, 1993, to make a 343°C+ (650°F+) hydrotreated make-up oil. The remainder of the start-up oil was hydrotreated and distilled as the reactor temperatures were raised to 385°C (725°F) during the initial coal operations. TNPS injection, used to presulfide the catalyst, was begun October 25 and terminated at 1615 hours October 28 after sulfur breakthrough in the vent gas was confirmed.

The recycle gas preheater control box and fuel gas control valve were replaced during start-up. Except for some minor adjustments to these controls, the heater operated well during the remainder of the run.

Valves in the common vent line between the two catalyst addition systems were found to be leaking on the process side during start-up, causing a slow pressurization of the opposite addition vessel during addition. These valves were repaired; however, future separation of the two vent systems all the way to the high pressure flare knockout is under consideration.

The slurry mix tank agitator shaft broke during start-up and was replaced.

The vacuum pump oil cooler was found to be broken. This was also replaced.

Periods 1-5

Period 1 commenced at 1300 hours October 29, 1993, when coal was introduced to the slurry mix tank. The first line-out condition was completed as scheduled in Period 2. Then in Period 3, the recycle slurry to coal ratio was lowered to 1.5 from 2.0, the first and second stage reactor temperatures were raised to 400°C (750 °F) and 427°C (800 °F), respectively, for the second line out condition. *Table 4.10* summarizes the operating conditions examined in this program.

In Period 5, the first and second stage reactors operating temperatures were being increased to 410°C (770 °F) and 435°C (815 °F), respectively, when at 1530 hours a high pressure tubing connection on the first stage catalyst withdrawal system failed. The unit was shutdown in an orderly fashion to repair this fitting. Examination revealed the coupling was not broken. However, the coned end of the line was damaged which led to the leakage.

Additional catalyst was added to the first stage in Period 2 and the second stage in Period 3. A pound or less of catalyst was withdrawn from each reactor in Period 1. At the start of Period 4, the projected catalyst inventories were 53 kg (117 lbs) and 55 kg (122 lbs) in the first and second stage reactors, respectively.

First stage catalyst addition was difficult throughout the first 5 periods. The associated addition vessel, O-15, and catalyst valves C and D were removed and inspected during this coal outage. The O-15 internal screen assembly was found to be broken, and pieces of the screen were plugging the outlet.

The unit was operated in an ashy recycle mode during Periods 1-5. Solids were rejected from the excess reactor liquid flash bottoms with the vacuum still bottoms stream. The remainder of the reactor liquid flash bottoms were recycled to accumulate solids and resid in the system. Hot separator overheads were sent to the in-line hydrotreater at 343°C (650 °F) to 357°C (675°F).

There were two brief interruptions in coal feed to the slurry mix tank during Periods 1 to 4. The first occurred after the P-2 coal hopper was refilled for the first time at 0203 hours of Period 1B, while the second occurred after P-2 was filled at 0712 hours of Period 4A. These outages lasted 6.33 hours and about 1 hour, respectively. During the first refilling of the coal hopper, 213 kg (469 lbs) of coal was added to the slurry mix tank within a 50 minute span. Apparently, the pneumatic transfer of coal from the storage bins (P-8, P-9) to the day coal hopper (P-2) provided sufficient force to fluidize coal through the screw feeder and into the mix tank. The second outage was caused by a blown fuse on the screw feeder.

The first solution to this situation was to close the knife valve at the end of the screw feeder while P-2 is being refilled. Starting the screw feeder with this knife gate closed probably caused the second outage (blown fuse). A procedural modification was next instituted redirecting the nitrogen purges away from the screw feeder. This allowed coal to be continuously fed to the slurry mix tank while P-2 was being filled. A rotary valve installed between the screw feeder and the coal hopper should provide a long term solution to this situation.

Periods 6-10

The first stage catalyst addition vessel was reinstalled after the internal screen was replaced with a splash tube. Operations were resumed after this vessel was pressure tested and insulated. This restart proceeded smoothly with the first and second stage reactors being ebullated November 5, 1993. Sufficient catalyst was added to each ebullated bed reactor, after ebullation was established, to raise the inventory in each reactor to the desired 57 kg (125 lbs).

TNPS was injected throughout the heat-up period to sulfide the fresh catalyst. Coal feed was resumed at 1500 hours on November 7, 1993. In general, the restart proceeded well. During Periods 6 - 10, the first and second stage reactors at 410°C (770 °F) and 435°C (815°F), respectively, processed 73 kilograms per hour (160 pph) of coal with 109 kilograms per hour (240 pph) of recycle material in the ashy recycle operating mode. Solids were removed from the unit in the vacuum still bottoms stream. Catalyst was added to and removed from each reactor without difficulty, per the run plan, during these periods. Special samples for Consol were taken in Period 9.

Several pipe unions in the ROSE-SR^{SM} section began leaking solvent after this section was heated in preparation for operation. These leaks were repaired during Periods 9 and 10. Commissioning the ROSE-SR^{SM} unit was further delayed to Period 11 after the top flange and the nuclear detector on the first settler were repaired. A solvent leak developed when the first stage settler temperature was raised in preparation for operation. This flange was cleaned, inspected, repaired and reinstalled. The nuclear detector, which earlier had a circuit board failure, developed a bad high meg resistor. A new resistor arrived November 12, 1993, and was promptly installed. Afterwards, this detector worked well.

The first stage sample system was off-line during these periods due a plug in the inlet to the checks.

At 1700 hours in Period 10, a loss of liquid level in the hot separator caused the unit to lose approximately 6.2 MPa (900 psi) in pressure. The rate of depressurization was sufficient to carry catalyst from the second stage reactor toward the hot separator. This catalyst restricted the transfer line between these two vessels. The unit was then shutdown to clean and inspect the second stage reactor as well as the hot separator. The following inspections and unit modifications were implemented before the unit was restarted.

- The second stage reactor bottom head (from the reactor originally operated at the Wilsonville pilot plant) was found to have a 13 cm (5 inch) long axial crack between the ebullating pump suction line and the 8 cm (3 inch) center hole. This was x-rayed and repaired.
- The transfer line between the first and second stage reactors contained a plug. This section of line was replaced.
- The hot separator let-down valve was slightly worn and found frozen in place by coal slurry. It is not known when this occurred.
- The automatic block valve tungsten carbide trim and seat located before the let-down valve were found badly scored. They were replaced.
- The second stage ebullating pump was found to contain a metal piece similar to a watch spring. Pieces of this had damaged the lipseal and had started to damage the bearings. Only the lipseal and the bearings were replaced.
- The first stage sample system was cleaned.
- Individual hydrogen purge controllers were installed on each hot separator pressure tap, replacing a common controller to three different pressure taps.
- Six 2-loop controllers were installed in the control panel and used for unit back pressure, make-up hydrogen, hot and cold separator level, reactor liquid flash drum level and the scrubber level.
- The hydrotreater alternative feed system was installed.
- A new nuclear gauge system was bought and installed on the hot separator. This 60 cm (2 foot) strip-type detector was used to monitor level as backup to the differential pressure transmitters.
- The three purge hydrogen pressure control loops were configured as "setpoint trim" loops, i.e., the operator establishes a local setpoint which the controller adds to another pressure in the unit to establish the set point for the purge pressure. This control scheme worked very well during this operation. These arrangements are:
    - first stage purge pressure was trimmed by the first stage recycle gas inlet pressure.
    - second stage purge pressure was trimmed by the second stage recycle gas inlet pressure.
    - the downstream purge pressure was trimmed initially by the unit back pressure, later it was trimmed by the second stage recycle gas inlet pressure.

The ROSE-SR^{SM} section was operated successfully during the Period 10 turnaround. This experience helped HRI to improve the operating and maintenance procedures while recovering oil from the unit vacuum bottoms material. ROSE-SR^{SM} unit operations in the 200 pounds per hour range were demonstrated. This corresponded to about 1 pound per minute of bottoms material or about the maximum rate expected during the first Proof of Concept operation.

Condition 1A, Periods 11-14

After the catalyst was ebullated, a total of 7.7 kg (17 lbs) of catalyst was added to the first stage reactor to bring this inventory back to the design. The Period 11 restart went very well with coal feed to the unit being resumed at 1157 hours on December 4, 1993. Recycle gas was directed through the fresh feed preheater after the transfer line between the recycle gas preheater and the reactor became restricted during Period 11. Apparently oil carbonized in one of the two check valves in this service. This routing of the recycle gas has been conducted successfully in previous campaigns without any ill-effects.

During Periods 11 and 12, the vacuum still was utilized to remove solids from the unit. At the end of Period 12, the first stage target temperature was 407°C (765 °F) and the second stage target temperature was 432°C (810 °F). Difficulty in reaching the second stage target temperature was experienced with a slurry to coal ratio of 2.0.

The transition to Condition 1A was accomplished by reducing the recycle rate of reactor liquid flash bottoms (ashy recycle) from 102 kg//hr (224 pph) to 54 kg/hr (120 pph) in Period 13. The ROSE-SR^{SM} unit was brought on line in Period 13. A portion of the reactor liquid flash slurry was recycled during Periods 11-14. Catalyst addition and withdrawal were conducted to the second stage reactor during Period 13 and the first stage in Period 14. The first and second stage reactor catalyst inventories were 57 kg (126 lbs) and 59 kg (130 lbs) respectively prior to the resumption of catalyst replacement. Unit operations including the ROSE-SR^{SM} section were smooth during these periods.

Condition 1B, Periods 15-19

The transition from Condition 1A to Condition 1B was accomplished by reducing the recycle rate of reactor liquid flash bottoms from 54 kg/hr (120 pph) to 35 kg/hr (77 pph) in Period 15A. The hydrotreater was taken off line at 1000 hours of Period 16 and was returned to service at 1600 hours of Period 17. This action was taken to obtain untreated distillate samples to monitor the hydrotreater performance.

At 2200 hours Period 15, the transfer of slurry from the hot separator, O-1, to the reactor liquid flash drum, O-13, became inhibited. Initial inspections indicated the level control valve became stuck in a near closed position. Coal feed to the slurry mix tank was discontinued at 2200 hours until unit conditions were stabilized at 0600 hours of Period 16. Unit conditions were gradually modified, returning the unit to the target operating conditions by the end of Period 16.

During Period 18, both hot separator level control valves were removed from the unit, cleaned, inspected and returned to service. Coal slurry feed to the unit was maintained during this period.

In both cases, the valve stems appeared to have insufficient travel to properly control the separator level. Further inspection revealed the mechanical balance bars, in both valve positioners, were not adjusted properly and were limiting stem travel.

ROSE-SR^{SM} Section Solvent Trim Cooler, M-27, became fouled in Period 17, apparently with resid or waxy material. The solvent temperature decrease had been reduced from about 55.5°C (100°F) to about 11°C (20 °F) since Period 11, and the temperature in the solvent holding drum O-67 was approaching 66°C (150 °F). Feed to the ROSE-SR^{SM} unit was suspended at 1445 hours to clean this shell and tube exchanger. Cooling water flow was then restricted to warm the exchanger and attempt to 'melt' the fouling material. This action was sufficient to return the cooler to its initial performance. Feed to the ROSE-SR^{SM} section was resumed at 1715 hours of Period 17 after returning to the target operating conditions. Solids were successfully removed from the unit by the ROSE-SR^{SM} process throughout Condition 1B except for this 2.5 hour outage.

Other than the few exceptions noted, the unit operated smoothly throughout Condition 1B at the target reactor conditions in the ROSE-SR^{SM} ashy recycle process mode.

<u>Condition 2, Periods 20-26</u>

The transition from Condition 1B to Condition 2 was accomplished by reducing the recycle rate of reactor liquid flash bottoms (ashy recycle) from 35 kg/hr (77 pph) to 0 and increasing the catalyst replacement rates from 0.125 kg/metric ton (0.25 lbs/ton) and 0.25 kg/metric ton (0.50 lbs/ton) in the first and second stage to 0.75 kg/metric ton (1.5 lbs/ton) and 1.5 kg/metric ton (3.0 lbs/ton), respectively.

The 260 unit operated smoothly throughout Condition 2 in the deashed oil recycle mode. Both ebullated bed reactors were operated at the targeted pressure, temperature and space velocity. The hydrotreater was on-line since about 1900 hours of Period 17. Solids were continually being successfully removed from the unit by the ROSE-SR^{SM} process.

The differential pressure between the first stage inlet and the back pressure controller gradually rose from about 860 kPa (125 psi) in Period 19 to 2070 kPa (300 psi) in Period 22. Water injection to the hydrotreater outlet line was increased to 30,000 cc/hour, and the associated line temperatures were raised to about 371°C (700 °F) at

0250 hours of Period 21. At 2055 hours of Period 22, the restriction was cleared, and the unit pressure drop returned to the 860 kPa (125 psi) range. The hydrotreater was taken off-line for a few minutes in Period 22 to determine if the pressure drop was in this reactor. Bypassing the hydrotreater had little or no effect on the unit differential pressure. The differential pressure between the first stage inlet and the unit back pressure again gradually rose from about 860 kPa (125 psi) in Period 22 to about 1380 kPa (200 psi) in Period 25 before returning to the 860 kPa (125 psi) range. These differential pressure buildups are normally experienced due to the formation of salts such as NH_4Cl in the high pressure piping.

A very dry powdery solids product was produced in Periods 23, 25 and 26. The ROSE-SR^{SM} unit was off-line twice in this reporting period. During Period 21A it was off-line for about 8.5 hours due to a pluggage of the first stage settler bottom outlet. The ROSE-SR^{SM} unit was taken off-line for about 2 hours in Period 23A to repair the solvent feed pump J-73. Each time the ROSE-SR^{SM} unit was returned to service without affecting the liquefaction operation.

The first stage ebullating pump seal oil pump was found off at 2150 hours of Period 23B. This pump was promptly restarted. The performance of the ebullating pump appeared to be consistent with its earlier performance, hence it did not appear that this seal oil outage had any detrimental effects.

Condition 3A, Periods 27-32

The transition Condition 3A began at 2300 hours of Period 27 and was completed by 1600 hours of Period 28. This condition change was delayed from the beginning of Period 27 due to mechanical issues in the ROSE-SR^{SM} unit. Included in this condition change were:

- 50% increase in space velocity
- 2.8°C (5 °F) increase in first stage reactor temperature to 410°C (770 °F)
- 5.6°C (10 °F) increase in second stage reactor temperature to 438°C (820 °F)
- taking the hydrotreater off-line at 0845 hours Period 27.

The ROSE-SR^{SM} unit had to be taken off-line at the start of Period 27 to repair block valves in the transfer line between the first stage settler and the bottoms receiver and to clear transfer lines leading to one of the ROSE-SR^{SM} feed pumps, J-72B. The block valves had become worn and contained residue on the seating service; both of these items prevented them from shutting tightly. Repairs were completed by 2000 hours, when procedures to restart the ROSE-SR^{SM} unit were initiated. Vacuum bottoms feed to the first stage settler resumed at 2237 hours of Period 27.

The fresh feed preheater air controller linkage became jammed at 0130 hours of Period 27, causing the preheater to loose temperature and the first and second stage reactor temperatures to drop to 376°C (708 °F) and 427°C (801 °F), respectively. This chain was tightened, and the preheater resumed normal operation shortly thereafter. The reactor temperatures were returned to their target values by 0300 hours.

Operations during Periods 29 through 32 were hindered by level control issues in the hot separator, cold separator and the scrubber. These level control problems and possible salt formation in the hot separator overhead line caused several fluctuations in the unit back pressure. These may have caused catalyst to be carried over into the hot separator.

Oil/water separation in the main oil-water separator O-45 was not complete, frequently sending water to the atmospheric still. The ROSE-SR^{SM} section was taken off line twice for cleaning and eventually both the vacuum still and the ROSE-SR^{SM} sections were shut down because insufficient material was being sent to the vacuum still to maintain operation in these areas.

Coal feed to the slurry mix tank was suspended at 0345 hours of Period 32 on December 26, 1993, after the hot separator letdown system quit passing material. Inspection of the hot separator, after the unit was shutdown, revealed the screen at the liquid inlet was collapsed and damaged at the point of entry into this vessel. It appears that the screen collapsed and inhibited flow out of the bottom of the separator.

Analysis of the oil/water separator operation indicates that, when most of the material in the hot separator was going overhead, the heavy hydrocarbons present in the overheads made separation difficult. Also the residence time in the water side chamber was reduced to 10 to 15 minutes. This low residence time was insufficient to perform the desired oil/water separation for this heavier oil/water mixture.

Between Periods 32 and 33 the settled catalyst bed heights for the first and second stage reactors were determined to be 2.9 m (9.5 ft) and 3.0 m (9.8 ft), respectively, by the nuclear gauges.

The hydrotreater bypass valve was observed to function correctly, indicating this valve was not the cause for the poor hydrotreater performance.

Several minor modifications/repairs were made to the unit between Period 32 and 33 including:

- replaced the worn Rockwell and Autoclave valves in the hot separator letdown system.
- replaced the blown rupture disc in the hot separator relief system.
- installed a larger naphtha stabilizer bottoms weigh tank so that naphtha pump outs would occur less frequently.
- cleaned and repaired the recycle gas preheater outlet line, then configured the piping so the recycle gas preheater is used to preheat recycle gas to the second stage reactor.
- installed a computer addressable make-up hydrogen compressor inlet meter and obtained additional calibration data on the make-up hydrogen orifice.

Periods 33-36

Coal processing resumed at 0600 hours on January 3, 1994. During Periods 33 and 34 the unit conditions were lined out at:

• First Stage Temperature	410°C (770 °F)
• Second Stage Temperature	432°C (810 °F)
• Space Velocity	480 kg/h/m^3 (20 lbs/hr ft)
• Solid Separations Mode	Vacuum still with ashy recycle
• Recycle:coal ratio	1.4 kg/kg (1.4 lb/lb)

The high pressure section operated smoothly during Period 34 and the first half of Period 35A, when the coal feed rate was increased to 400 kg/h/m^3 (25 lbs/hr/ft^3). At 1435 hours of Period 35, the right side hot separator level control valve trim broke. Over the next three hours at least four hot separator letdown valves appeared to become jammed. At 1700 hours, the coal feed rate was reduced to 320 kg/h/m^3 (20 lbs/h/ft^3), and these liquefaction conditions were maintained throughout the remainder of Period 35.

During Period 36, the current set of 260 unit letdown trims were measured and compared to the 227 unit stock of letdown trims. The current batch of trims was slightly larger in diameter (by about 0.0025 cm (0.001 inch)) than previous trims. This slight change reduced the width of the gap between the trim and the seat by about 50% (from about 0.005 cm (0.002 inch) to 0.0025 cm (0.001 inch)). After this information was obtained, trims from the 227 unit stock were installed in the 260 unit letdown valves and moves were begun to increase the coal feed rate to the desired 300 kg/h/m^3. This increase in feed rate was underway when the O-5 level control valve trim broke at 1823 hours January 6, 1994. The unit back pressure fell to approximately 13.8 MPa (2000 psig) before the broken valve was blocked in. This sudden pressure drop appears to have caused excessive catalyst carryover into the hot separator. Coal feed to the unit was suspended at 1845 hours. Shutdown commenced shortly thereafter, once it became apparent that the hot separator was not going to recover from this upset.

The ROSE-SR^{SM} section was operated throughout Period 34. It was shutdown at 0830 hours of Period 35 after the gate valves on the bottom of the first stage settler would not seal. These valves were replaced, and the associated lines were cleared, before the ROSE-SR^{SM} was returned to service at 0655 hours of Period 36. The ROSE-SR^{SM} Unit was then shutdown at 1000 hours after the newly installed gate valves became cut and would not seal.

After Period 36, the hot separator, vacuum still and the first stage settler were inspected. The hot separator screen was partially collapsed with about 2/3 of the screen restricted by catalyst. This separator was cleaned, the screen repaired and the vessel reassembled. A new hot separator letdown trim design was developed, and trims consistent with this design were installed in the hot separator and cold separator letdown systems.

Approximately 57 liters (15 gallons) of hard, carbonaceous material was removed from the vacuum still. The origin of this material is unknown, since it has been several years since the last time this still was disassembled. A set of corrosion coupons supplied by Oak Ridge National Lab was installed in the vacuum tower when it was reassembled.

New Mogas block valves were installed upstream of the ROSE-SR^{SM} first stage settler level control valves, replacing the worn gate valves which cut rapidly and were prone to material build up on the seat.

The sample system was cleaned again and returned to service.

Periods 37 and 38

Operations in Period 37 and 38 were interrupted by mechanical failures causing unit upsets and additional downtime. At the end of Period 37, January 15, 1994, the hot separator relief valve opened and would not reseat causing the unit to rapidly depressure. Emergency procedures were used to shut the unit down and to flush the reactors. It appears that although inconel was specified for the upstream rupture disc, a stainless steel disc was delivered and installed. Fragments of this disc were found inside the relief valve, preventing the valve from reseating.

Although the unit could not be repressurized above 690 kPa (100 psig), both catalyst beds were promptly re-ebullated and liquid flows were maintained through the reactors and the downstream equipment while the reactors were cooled down. Amazingly, very little catalyst was displaced during this depressurization as evidenced by the settled catalyst bed heights and inspections of the hot separator and the hot flare drum.

The relief valve in question was placed in service as part of the hydrotreater installation, since the hydrotreater valving would allow an operator to block in the high pressure section of the unit. Because of the extent of damage to the relief valve, it would have taken one to two weeks to get the relief valve in question repaired. Modifications to the hydrotreater bypass valve were made to ensure this valve can not be shut. This allowed coal operations to be resumed promptly rather than wait for this valve to be repaired.

At 0315 hours of Period 37B, approximately 141 kg (310 lbs) of coal appeared to have been transferred into the slurry mix tank while the day coal hopper was being filled with coal. This was the first time since Period 4 that coal was rapidly transferred to the slurry mix tank while the day hopper was being filled. Extra oil was added to the slurry tank, and coal feed to the slurry mix tank was suspended until the slurry viscosity returned to normal. This incident accentuated the need for a rotary valve on the outlet of the day hopper, but in no way was this event thought to be related to the relief valve failure.

Coal feed to the unit resumed at 0400 hours on January 21, 1994, in Period 38, utilizing the vacuum still for solid rejection in the ashy recycle mode of operation. Approximately eight pounds of catalyst was added during this start-up to each reactor to return each reactor's catalyst inventory to the target amount. At approximately 2217 hours on January 21, 1994, the ebullating oil flow to the first stage reactor was lost. It appeared that a quantity of catalyst got into the ebullating pump and restricted the pump discharge line. Several events were occurring simultaneously which would tend to lift the catalyst bed, including an increase in the slurry feed coal concentration due to a pluggage in the recycle oil line, the catalyst bed was high and increases in gas rates were being made to compensate for consumption. Although decreases were made in the ebullating pump speed control, it does not appear that they were sufficient or made quickly enough to keep the catalyst bed at the proper height.

The first stage reactor was disassembled, cleaned and reassembled after Period 38. It was then recharged with the catalyst recovered from it. An additional 3.6 kg (8 lbs) of first stage catalyst removed during Period 37 was added to bring the first stage reactor inventory up to 90% of the run target prior to the restart.

Condition 3B, Periods 39-44

Coal operations were resumed at 1200 hours on January 29, 1994. Operations throughout the Period 39 restart were smooth. The vacuum still was utilized in Periods 39-41 to remove solids from the unit. Reactor flash liquid was recycled during Periods 39 and 40 (ashy recycle operating mode). Naphtha stabilizer bottoms collection, for outside studies, began in Period 40. Unit conditions were adjusted to 400 kg/h/m^3 (25 lbs/h/ft^3) , 1.2 recycle to coal ratio with 410°C (770 °F) and 432°C (810 °F) first and second stage reactor temperatures in Period 41, the start of Condition 3B.

A 1516 gram first stage reactor liquid sample was taken in Period 40. The impulse pump diaphragm broke in Period 41 allowing the hot check inlet to become restricted. However, the material in the sample vessel was isolated and collected. These were the only first stage samples obtained during this run.

Operations throughout Periods 42, 43 and 44 were smooth prior to 1633 hours on February 3, 1994, when ebullation was interrupted in both reactors while catalyst addition was being performed to the first stage reactor. Ebullation of the second stage reactor was regained at 1715 hours, while the first stage reactor was again ebullated at 0815 hours on February 4, 1994. It appears catalyst was carried into the first stage pump suction, interrupting this flow and causing the first reactor to degas. The second stage ebullating pump lost suction when the second stage liquid level fell below the suction cup due to the first stage reactor problem. Oil flows were not stopped during this coal outage.

The second stage addition vessel became plugged in Period 44 and would have had to be removed from the unit to be cleaned. However, the decision not to do any additional second stage catalyst replacement in this run was made, rather than to unplug this vessel. A repair would have required a unit depressurization and an extended coal outage.

The ROSE-SR^{SM} section began operation at 1525 hours of Period 42. There were two minor outages during this reporting period. The first outage was during Period 43 for 3.5 hours due to a restriction in the solids receiving vessel. The second outage lasted about 2 hours during Period 44 and was due a first stage settler feed pump pressure switch failure.

The flare system flame arrester (detonator type) became restricted in both Periods 43 and 44. This was cleaned each time, and the flare was returned to service. During each outage the bench unit flare system was utilized. The approximately 15 meter (50 ft) vertical line between the flare and the flare knockout drum condenses volatiles, which were causing the flame arrester to become restricted. The arrester was relocated closer to the knockout, and regular low point draining of this line has been instituted. No additional restriction have occurred in the flare header since these modifications were made in Period 46.

Condition 4A, Periods 45-48

Coal was reintroduced to the unit at 0400 hours on February 5, 1994, the start of Period 45. Ashy recycle was used during Periods 45 and 46 to increase the solids loading in the unit. Unit operations were modified in Period 47 to 400 kg/h/m^3 (25 lbs/h/ft^3), 1.1 recycle to coal ratio with 410°C (770 °F) and 432°C (810 °F) first and second stage reactor temperatures. Periods 47 and 48 were considered Condition 4A. In general, the unit operated smoothly during Periods 45-48.

The first stage catalyst addition line was restricted. Attempts to clear this line were made in Periods 46, 47 and 48. The first stage addition line was cleared in Period 51.

The ROSE-SR^{SM} section continued to operate smoothly throughout Periods 45-48.

Condition 4B, Periods 49-51

The transition to Condition 4B (400 kg/h/m^3 (25 lbs/h/ft^3) and a 1.0 oil:coal ratio) began at 1200 hours of Period 49 and was completed within 24 hours.

Coal processing was suspended for 38 hours beginning 0300 hours on February 11, 1994, Period 50 after the make-up hydrogen rate was increased by 25%. This caused the first stage ebullating pump to lose suction and the catalyst bed to slump. Ebullation was regained at 1040 hours on February 11, 1994. The resumption of coal processing was delayed because the hydrogen supplier could not guarantee delivery before February 13 due to a heavy winter snow storm. During this coal outage (counted as Period 51), 5300 liters (1400 gallons) of recently obtained gas oil was hydrotreated between 1400 hours on February 11 and 1700 hours on February 12, 1994. The reactor temperature for the first 21 hours was 349°C (660 °F); it was then increased to 391°C (735 °F) in preparation for coal cut-in. This oil now is available as make-up oil.

Condition 4C, Periods 52-57

Coal feed was reintroduced to the unit at 1700 hours on February 12, 1994, in Period 52. Ashy recycle was used during Periods 52 and 53 to increase the solids loading in the unit. Unit conditions were adjusted to achieve 481 kg/h/m^3 (30 lbs/h/ft^3) and a 0.9 oil: coal ratio in Period 55B. This mixture pumped well; however, at the 481 space velocity conditions it caused an approximately 1 MPa (150 psi) pressure drop across the preheater. A preliminary review of this situation indicated that this pressure drop was due to the volume and viscosity of the material being processed and not due to any buildup in the coil. The oil-to-coal ratio was raised to 1.2 at 1915 hours of Period 56, because the combination of salt buildup in the hot separator overheads line and the high preheater coil pressure drop was making it hard to get make-up hydrogen into the unit. Condition 4C was considered to be Periods 54-57.

The ROSE-SR^{SM} section continued to process material until 0630 hours of Period 51, when it ran out of feed material due to the coal outage. ROSE-SR^{SM} operations resumed at 1600 hours of Period 53 after sufficient VSBs had been accumulated in O-60 and O-61. The ROSE-SR^{SM} restart went very well.

At 0930 hours of Period 54, approximately 164 kg (362 lbs) of coal was added to the slurry mix tank during a scheduled filling of the coal weighing hopper. Additional coal feed to the mix tank was suspended until the slurry mix tank viscosity returned to the value it was before this incident. It appears there were no long term effects from this incident.

Additional pressure drops as high as 1 MPa (150 psi) were observed in the hot separator overheads line. This appeared to be caused by salt build-up in this line due to the low water injection rate of 0.2 kg water/kg of coal feed. These pressure drops were controlled by switching the water injection port every two to four hours. This situation was tolerated because the current oil-water separator could not process the proper amount of water and keep water out of the atmospheric still.

Condition 5, Period 58

Special testing of the filter, the ebullated bed reactors and the alternate hydrotreater feed system were conducted in Period 58. Reactor bottoms flash liquid was accumulated in Period 58 to perform special filtration tests. Two filter cycles were completed. It appears, from the pressure drop data and the analytical data, that there was bypassing of the filter leaves, possibly caused by a failed gasket. Inspection of the filter gaskets confirmed the gaskets were damaged. They may have dried out since the filter was reassembled prior to Period 1.

The alternate hydrotreater feed system was tested in Period 58. It proved to be capable of heating reactor liquid flash vessel overheads up to 371°C (700 °F) and pumping them, as they are produced, into the hydrotreater.

The ROSE-SR^{SM} section continued operation until it ran out of feed at 2150 hours of Period 58. This section operated whenever there was feed available after Period 43. Analysis of the bottoms indicated an increase in the asphaltene content in the ROSE-SR^{SM} bottoms over the last couple of weeks of the run.

Shutdown

Run 260-04 was completed at 0600 hours on February 19, 1994. The 260 unit was shutdown after processing Illinois No. 6 coal for 58 days.

D.5 Unit Inspections

The vacuum still, hot separator, cold separator, reactor liquid flash drum, hydrotreater, clean oil tank, recycle weighing tank, the two catalyst addition vessels and the ebullating pumps were inspected during this shutdown. The vacuum still, hot separator and the reactor liquid flash drum were clean. Approximately one gallon of carbonaceous sludge was in the cold separator. This is normal.

The hydrotreater internals were intact. The vessel walls were dry on the top half of the vessel and oil-soaked around the bottom half. A minimal amount (less than a cubic inch) of carbonaceous material was found on each of the two distributors and on each of the two inlet screens. Approximately a gallon of fine dry powdery carbonaceous material was found under the inlet screens above the inlet packing. The gasket around the top catalyst basket was intact while the gasket around the bottom basket was missing. Samples of each catalyst bed (top and bottom) and each mass of fine dry powdery carbonaceous material were taken.

The clean oil tank contained about 8 cm (3 inch) of carbonaceous sludge, while the recycle weighing tank had closer to 15 cm (6 inch) of similar material.

The ebullating pumps were disassembled and sent to Dixon Products for detailed inspection which will be summarized separately. All rotating parts in both pumps rotated freely. Solids were found to have gone past the lipseal in the first stage reactor pump. The stationary parts (e.g., the front bearing housing) were very hard to remove and are discolored by a green or whitish film. This film appears to hinder parts removal, but only appears on the stationary parts and not on the rotating parts.

The first stage catalyst addition vessel was clean and mechanically intact. The second stage catalyst addition vessel was also clean, but the splash tube inside the vessel was collapsed blocking any entrance to the vessel. The tube specifications are being upgraded from schedule 10 to schedule 80.

D.6 Procedural and Unit Modification Suggestions

1. When commissioning the ebullating pump seal oil system, the ebullating pump casing should be bled down to remove any gas from the pump.

2. Pulsation dampeners should be installed on the ebullating seal oil pumps.

3. Brooks mass flow controllers used to control the high pressure gas purge streams, work very well when clean; however, a small quantity of oil will put the meter out of service. Frequently (especially during start-up), these meters become fouled with oil, either from the compressor or the process. Coalescers have been purchased and need to be installed both upstream and downstream of these controllers to minimize oil contamination.

4. O-46 is used as a reactor liquid flash weigh tank. Material from this tank goes to the filter, the vacuum tower feed accumulator, the recycle oil blending tank or the ROSE-SR^{SM} unit depending on the PDU operating mode. Currently, the inventory in this vessel is monitored by level indication only. Putting this tank on a weigh cell will greatly increase blending accuracy and decrease the effort needed to convert from level to weight.

5. The vacuum still overhead and the sour water scales are BCD type-equipment which were never connected to the revised Process Control system. These scales are over 15 years old and need to be replaced with 1-5 volt systems and connected to the process control system. Originally, HRI had about 10 of these BCD-type scales. All of the others have been replaced.

6. The recycle gas compressor vent currently discharges to the outdoors. This vent needs to be connected to the flare relief header.

7. Prior to POC-1, the recycle gas heater was used to preheat recycle gas to the first stage reactor, and electrical resistance windings were used to preheat recycle gas to the second stage reactor. During POC-1, the transfer line between the recycle gas heater became restricted during the Period 6 and 11 restarts, forcing routing of this recycle gas to the fresh feed preheater. Later in the run, it became apparent that windings were not going to provide adequate heat to the second stage recycle gas to achieve the desired reactor temperatures. We then rerouted the second stage recycle gas through the recycle gas heater. This arrangement has worked well and provided good reactor temperature control. However, the Autoclave check, which became restricted twice during this operation, is still in the heater discharge line. We

expect to eliminate this restriction by replacing this check valve with a high temperature Mogas ball valve.

8. The transfer of high solids containing material from the ROSE-SR^{SM} first stage settler to either of the two bottoms receivers has been a difficult task during POC-1. A slight upset and this line becomes restricted. We have already replaced the upstream gate valves with Mogas block valves. These valves have worked well providing a tight shutoff between the settler and the receiver. Additional changes needed to this section include:

   - Installing an upstream (before the LCV) gas oil flush. This will allow flushing through the LCV into the receiver.
   - Installing a normally open gate valve just downstream of the Mogas block valve. This will allow for higher pressure purging through the LCV into the receiver, since the Mogas valve is a unidirectional valve.
   - Relocating the LCV closer to the receiver.
   - Installing a permanent vso purge system. Currently, operators hand carry gas oil to a local purge pump. This offers a limited supply, is inconvenient and increases the likelihood of oil spills.

9. The main oil water separator in the PDU was designed to handle approximately 40 kg/hr (88 pph) of water and 54 kg/hr (120 pph) of oil. The current POC unit configuration with the reactor liquid flash overheads being recycled to the hydrotreater calls for water and oil rates of 105 kg/hr (232 pph) water and 156 kg/hr (344 pph) oil at a 136 kg/hr (300 pph) coal feed rate. The current design cannot handle these rates and provide reasonable oil/water separation.

10. The first stage reactor sample system has worked very well in the bench units. Two first stage reactor liquid samples were obtained during POC-1. Additional samples would have been taken, if we have been able to purge the associated hot check suction line. We believe having a purge oil line installed to clear restrictions on the hot check inlet should allow for several first stage samples to be taken in the next POC run.

11. The ROSE-SR^{SM} unit utilizes a hot/cold solvent exchanger. This exchanger works well when the unit is lined out. However, it tends to snowball unit upsets. If the returning solvent is too hot, it will heat the feed solvent too much; if the returning solvent is too cold, it will not heat the feed solvent enough. Use of a Dowtherm heated trim heater downstream of the current exchanger will ensure the feed solvent will be at the desired temperature before it mixes with the resid stream.

12. Currently atmospheric bottoms go to the recycle oil blending tank, and excess vacuum overheads go to the clean oil tank or tank farm. We desire to switch these two streams so that any heavy oil returned to the tank farm is hydrotreated. This is primarily a piping change, but it includes a few additional windings and temperature control loops, since the vacuum still overhead line will now be longer.

13. The flare is currently located on the gasifier tower. The burner is contained within a thin metal shroud. This shroud gets very hot and glows red at night. A thermocouple is attached to the shroud and used to monitor the flare flame temperature. This thermocouple currently fails within a week, as it is in direct contact with the flame, causing the metal sheath to melt. Our air permit requires a flame temperature of 925°C (1700 °F) be maintained to ensure proper combustion of materials. We propose to:

    - line the inside of the shroud with a ceramic liner,
    - install a thermowell to protect the thermocouple, and
    - upgrade this Type J thermocouple to a higher temperature thermocouple.

14. Each of our ebullated bed reactors has 10 internal, side-entry thermocouples. These thermocouples are all attached to the reactor via threaded stainless steel fittings. Each time the ebullating oil cup is removed from the reactor, these thermocouples have to be removed. With time these fittings become galled, unusable and require repair. We are proposing to design a standard repair which will include a Grayloc-type connection and having parts for three repairs maintained inventory. Repairs will be done on an as-needed basis.

15. Hydrotreater process performance during the first POC operation indicated material was bypassing the catalyst baskets. A new configuration of the hydrotreater internals has been designed, which does not include catalysts baskets and, therefore, should not allow bypassing of the catalyst.

16. The new coal handling system pneumatically conveys material from the long term storage bins to our day hopper (P-2). Coal is then transported by a screw conveyor into the slurry mix tank. If the conditions in the day hopper are correct, large quantities of coal are transported into the slurry mix tank when the day hopper is being filled. This phenomena can shut down the PDU, if the coal loading in the slurry mix tank is allowed to increase excessively. Installation of a rotary valve between the day hopper and the screw conveyor should stop this, since it would serve as a pressure barrier and prevent the pneumatic transfer of coal through the screw.

17. O-44 is an oil/water separator downstream of the primary oil/water separator. Water from our primary separator is decanted in this glass vessel to improve oil recovery. This vessel ruptured during the first POC run, when the flare header became restricted. It must be replaced with a carbon steel vessel for safety reasons.

18. The separator obtained from Wilsonville was used in the first POC operation because it was larger in diameter than the previously used separator. Both separators have a smaller diameter liquid section and a larger diameter vapor section. However, the inlet to the Wilsonville separator is in the 8 cm (3 inch) bottom section. It should be in the 15 cm (6 inch) vapor section. Relocating this inlet will reduce entrainment of heavy materials into the cold separator and oil/water separator.

19. O-36 is the naphtha stabilizer feed accumulator. It a small 15 cm (6 inch) diameter vessel, primarily used as a sight glass. It needs to be replaced with a metal vessel for safety reason. The new vessel also needs to have a water boot so that the entry of water into the naphtha stabilizer can be prevented.

20. In the past HRI has added catalyst through the top of the reactor. The current PDU reactors have side-entry catalyst addition nozzles. These nozzles were incorporated into the design of these vessels because the Rockwell plug valves, originally used for catalyst handling, were very large. Top loading of catalyst into the current ebullated bed reactors with Rockwell valves would have exceeded the local township zoning. We believe the current catalyst addition system with the Valvtron dual valves can be installed without exceeding the local zoning laws. The side-entry nozzles appear to have become restricted during the first POC campaign. Cold modeling studies indicate that a portion of these side-entry nozzles are liquid/catalyst filled at all times. We believe this restricts the flow of fresh catalyst into the main portion of the catalyst bed. HRI is proposing to plug the side-entry nozzles and add catalyst through the top of the reactor.

21. The fresh feed preheaters developed a 0.7 to 1 MPa (100 to 150 psi) pressure drop when the unit was operated at a 0.9 oil to coal feed ratio. We plan to install a larger diameter coil for this preheater.

TABLE 4.1

ANALYSIS OF ILLINOIS NO. 6 COALS

HRI No.	6081*	6125*	6141**
Mine:	Burning Star No. 2	Burning Star No. 4	Crown II
Proximate Analysis:			
Moisture, W%	2.97	8.05	15.18
Volatile Matter, W% dry	39.05	41.67	41.88
Fixed Carbon, W% dry	50.34	50.07	48.68
Ash, W% dry	10.61	8.25	9.44
Ultimate Analysis, W% maf			
Carbon	77.73	76.96	77.74
Hydrogen	4.91	5.21	5.67
Nitrogen	1.40	1.53	1.70
Sulfur	4.12	3.52	4.37
Oxygen (by diff.)	11.85	12.78	11.41
Chlorine, W%	0.18+	0.04	0.14
Mineral Analys, W% Ash	(+)	(+)	
SiO3	45.5	48.0	47.9
Al2O3	17.0	19.1	17.3
TiO2	1.0	0.9	0.9
Fe2O3	19.2	20.0	19.1
CaO	8.5	5.0	5.0
MgO	1.0	0.8	0.9
Na2O	0.9	0.5	1.4
K2O	2.8	2.0	1.9
P2O5	0.1	0.2	0.2
SO3	1.6	2.3	4.8
Undet.	2.4	1.2	0.7

* HRI data
** Commercial Testing & Eng. Co.
\+ Report DOE/PC 89883-23

Table 4.2

Petrographic Analyes of Candidate Illinois Feed Coals
(Ref: Wilsonville Run No. 257 Report)

Mine Name	Crown II	B.S. # 4	B.S. # 2
Wilsonville #	9855	10012	87927
Mean Max Reflectance (Ro), %	0.48	0.51	0.53
<u>Maceral Analysis, Vol %,</u> <u>(Mineral Matter Free Basis)</u>			
Reactives	96.3	92.5	94.0
Vitrinite	90.2	87.4	89.0
Type 3	1.7	0.0	0.0
Type 4	61.3	38.5	30.3
Type 5	27.1	45.4	42.6
Type 6	0.0	3.5	16.0
Exinite	5.3	4.1	3.7
Resinite	0.0	0.0	0.0
1/3 Semifusinite	0.7	1.0	1.4
Inerts	3.7	7.5	5.9
2/3 Semifusinite	1.5	1.9	2.8
Micrinite	1.1	2.0	1.7
Fusinite	1.2	3.5	1.4
Mineral Matter, Vol %	6.1	6.9	7.6

TABLE 4.3.

GRINDING RESULTS USED FOR PROCESS CONTROL
(recorded while HRI was present)

	30 min * into test	60 min * into test	50 min into run	2 hours into run	3 hours into run
Sieve Analysis, Mesh Size (wt%)					
>40	0.4	0.0	0.0	0.0	0.0
40-50	0.0	0.0	0.0	0.0	0.0
50-200	4.4	3.6	4.8	2.8	8.0
200-325	62.4	61.0	61.2	41.8	56.4
<325	32.8	35.4	34.0	55.4	35.6
Moisture, (wt%)					
30 min	3.6				
60 min				2.6	
90 min	3.2	4.0			
* These samples are before the unit is purged.					

TABLE 4.4

GRINDING RESULTS USED FOR PROCESS CONTROL
(recorded while HRI was not present)

Time	9/15 4:00	9/15 4:30	9/16 8:00	9/16 9:30	9/16 11:00	9/16 12:30
Sieve Analysis, Mesh Size (wt%)						
>40	0.0	0.0	0.0	0.0	0.0	0.0
40-50	0.0	0.0	0.0	0.0	0.0	0.0
50-200	10.0	9.8	12.0	10.8	8.0	6.0
200-325	66.6	58.4	52.8	55.4	70.8	59.2
<325	23.4	31.8	35.2	33.8	21.1	34.8
Moisture, (wt%)						
1 hour		3.6	5.4		5.6	4.8

TABLE 4.5

COAL ANALYSIS

HRI-#	HRI-6156	HRI 6157
SAMPLE TAKEN	Taken by HRI at Empire Coke 1 hour into grinding	Taken by HRI at Empire Coke from feed to mill
SAMPLE TYPE	Ground Coal	Raw Coal
MOISTURE (wt%)	3.39	
ULTIMATE ANALYSIS (wt% dry basis)		
CARBON	71.2	
HYDROGEN	5.25	
SULFUR	4.02	
NITROGEN	1.42	
ASH	10.24	
OXYGEN (by diff)	7.87	
SULFUR FORMS (wt% dry basis)		
SULFATE	0.01	0.01
PYRITIC	1.08	1.06
ORGANIC	2.93	2.89
TOTAL	4.02	3.96
MINERAL ANALYSIS (wt% of ash)		
PHOSPHORUS	0.23	
SILICON	49.98	
IRON	16.34	
ALUMINUM	18.64	
TITANIUM	0.95	
CALCIUM	4.05	
MAGNESIUM	0.87	
SULFUR TRIOXIDE	3.38	
POTASSIUM	2.27	
SODIUM	1.48	
STRONTIUM	0.03	
BARIUM	0.01	
MANGANESE	0.10	
UNDETERMINED	1.67	
TOTAL	100.00	
MISCELLANEOUS ANALYSIS (dry basis)		
HEATING VALUE (btu/lb)	12716	
CHLORINE (wt%)	0.11	0.13
BASE/ACID RATIO	0.36	
FOULING INDEX	0.53	
SLAGGING INDEX	1.45	

TABLE 4.6

EMPIRE COKE ANALYSIS OF GROUND COAL BATCHES

Date	9/16/93	9/21/93	9/27/93	10/4/93
Run #	267	269	271	273
% Moisture	5.82	5.02	6.99	4.16
% Volatile	41.40	41.55	41.22	41.21
% Fixed Carbon	49.18	48.73	48.44	49.07
% Ash	9.42	9.72	10.34	9.72
% Sulfur	4.02	3.98	4.04	3.92

TABLE 4.7
INSPECTION OF CANDIDATE STARTUP/MAKE UP OILS FOR P0C-01

HRI No.	L-800	L-803	HRI-6172	Filtered L-769	LCO:HRI-5669
API Gravity	9.6	12.2	8.5	19.4	18.3
Elemental Analysis, W%					
Carbon	87.85	88.47		88.38	86.94
Hydrogen	9.86	10.54		11.56	9.8
Sulfur	1.93	0.83		0.09	0.3
Nitrogen	0.22	0.11		0.07	0.038
ASTM D-1160 Distillation, Deg. C					
IBP	219	307	186	253	198
5 V%	287	339	314	277	247
10 V%	315	346	359	293	258
					274
20 V%	343	372		308	283
30 V%	373	387	406	321	292
40 V%	391	399		334	301
50 V%	410	412	434	343	311
					323
60 V%	429	428		362	337
70 V%	445	445	465	378	357
80 V%	462	458		398	369
90 V%	512	498	519	439	
91 V%	524		538	467	
Weight Percents					
IBP-343 Deg. °C	18.15	6.19		47.87	
343-454 Deg. °C	52.24	65.58		42.64	
454-524 Deg. °C	18.35	18.07		6.08	
524^{+} Deg. °C	10.57	9.64		2.56	
Loss	0.69	0.52		0.86	
% Aromatic Carbon	44.5	32.1			
% Cyclic Hydrogen	22.11	28.4			

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TABLE 4.8
INSPECTIONS OF STARTUP/MAKEUP OIL

	Aromatic		Cyclic		Paraffinic		
	Condensed	Uncond.	Alpha	Beta	Alpha	Beta	Gamma
POC-01							
Untreated (L-800)	7.28	6.47	8.09	14.02	9.16	29.11	25.87
Treated (L-803)	3.22	5.36	9.38	19.04	8.31	30.83	23.86
PDU 260-3							
S/U Oil	6.7	5.8	13.5	20.7	9.1	27.1	17.1
ShutDown	4.9	3.9	10.3	20.0	7.7	34.3	19.0

TABLE 4.9

Comparison of Physico-chemical Properties of AO-60 Catalyst Used for POC-01 with S-317 Catalyst
Used for POC-01 with S-317 Catalyst

CATALYST	Akzo-AO-60	Shell S-317
Nominal Size	1/16"	1/32"
W% Molybdenum	12.25	10.76
W% Nickel	2.6	2.86
Bulk Density, g/cc	0.57	0.6
Particle Density, g/cc	0.87	0.99
Surface area, m2/g	286	263
Pore volume, cc/g	0.874	0.681
Avg. Crush strength, lb/m	2.67	1.78
Avg. Diameter, mm	1.59	1
Avg. Length, mm	3.8	3.8
Avg. Pore diameter, Ao	125	105
PERFORMANCE: W% MAF		
Run No.	CC-16	CTSL
524 C^+	89.7	87.7
C4-524 C	72-74	72-76

TABLE 4.10
ACTUAL RUN CONDITIONS FOR POC-01 (RUN 260-04)

PERIODS	COND	SPACE VELOCITY kg/hr/m^3 (lb/hr/ft^3)	REACTOR TEMP,°C (°F) K-1	REACTOR TEMP,°C (°F) K-2	SOLV/COAL RATIO	SOLIDS REMOVAL	CATALYST REPLACEMENT RATE kg/metric ton (lbs/ton) K-1	CATALYST REPLACEMENT RATE kg/metric ton (lbs/ton) K-2
1-2	L/O	320 (20)	390 (735)	413 (775)	2.0	VAC STILL	0.25 (0.5)	0.50 (1.0)
3-5*	L/O	320 (20)	399 (750)	427 (800)	1.5	VAC STILL	0.25 (0.5)	0.50 (1.0)
6-7	L/O	320 (20)	410 (770)	435 (815)	2.0	VAC STILL	0.25 (0.5)	0.50 (1.0)
8-10*	L/O	320 (20)	410 (770)	435 (815)	1.5	VAC STILL	0.25 (0.5)	0.50 (1.0)
11-12	L/O	320 (20)	407 (765)	432 (810)	1.5	VAC STILL	0.25 (0.5)	0.50 (1.0)
13-19	1	320 (20)	407 (765)	432 (810)	1.2	ROSE	0.25 (0.5)	0.50 (1.0)
20-26	2	320 (20)	407 (765)	432 (810)	1.2	ROSE	0.75 (1.5)	1.50 (3.0)
27-32*	3A	480 (30)	410 (770)	438 (820)	1.2	ROSE	0.75 (1.5)	1.50 (3.0)
33-36*	L/O	480 (30)	410 (770)	432 (810)	1.2	VAC STILL	0.75 (1.5)	1.50 (3.0)
37*	L/O	480 (30)	410 (770)	432 (810)	1.2	VAC STILL	0.75 (1.5)	1.50 (3.0)
38*	L/O	480 (30)	410 (770)	432 (810)	1.2	VAC STILL	0.75 (1.5)	1.50 (3.0)
39-40	L/O	320 (20)	410 (770)	432 (810)	1.2	VAC STILL	0.75 (1.5)	1.50 (3.0)
41-44*	3B	400 (25)	410 (770)	432 (810)	1.2	ROSE	0.75 (1.5)	1.50 (3.0)
45-46	L/O	320 (20)	413 (775)	432 (810)	1.2	VAC STILL	---	---
47-48	4A	400 (25)	413 (775)	432 (810)	1.1	ROSE	---	---
49-50	4B	400 (25)	413 (775)	435 (815)	1.0	ROSE	---	---
51-53	L/O	320 (20)	413 (775)	432 (810)	1.2	VAC STILL	---	---
54-57	4C	480 (30)	413 (775)	435 (815)	0.9	ROSE	---	---
58	5	480 (30)	413 (775)	435 (815)	0.9	FILTER	---	---

Notes: Regular catalyst additions and withdrawals were suspended after Period 44. Catalyst was added to the first stage reactor after Period 51 to adjust inventory.

* Unit shutdown at the end of these periods.

TABLE 4.11

POC-01 (RUN 260-04)
OVERALL MATERIAL BALANCE FROM END OF RUN

	TOTAL KILOGRAMS
STREAMS IN	
Coal Feed (Wet)	101821
Make-up Oil to SMT	30491
SMT Inventory Loss	-675
Ebullating Seal Oil	3436
Make-up Oil, Purge	1218
H_2O to O-1	35625
Fresh Hydrogen	7800
DMDS (TNPS	9
Make-up Solvent, ROSE	0
TOTAL FEED	179725
STREAMS OUT	
Vent Gas (H_2O & N_2 Free)	2357
BTMS Gas (H_2O & N_2 Free)	8534
SMT Vent Drain	114
Unit Knockouts	2742
Naphtha Stabilizer Bottoms	58732
ASB Products	3270
Separated Water	47186
VSO Product	3293
VSB Product	16424
Pressure Filter Cake	0
Filter Inventory Change	0
ROSE DAO Product	229
ROSE Bottoms	15508
ROSE Inventory Change	2522
Recycle Oil Inventory Change	1207
Vacuum Still Inventory Change	-348
RLFVB Inventory Change	2358
TOTAL PRODUCT	164129
OVERALL BALANCE %	91.27

TABLE 4.12

POC-01 (RUN 260-04)
LIQUEFACTION BALANCE FROM END OF RUN

	TOTAL KILOGRAMS
STREAMS IN	
Coal Feed (Wet)	101821
Recycle to SMT	102087
Make-Up Oil to SMT	30491
VSOH to SMT	25610
SMT Inventory Loss	-675
Ebullating Seal Oil	3437
VSO To Purge Pump	11954
Make-Up Oil, Purge	1218
H_2O Injected to 0-1	35625
Fresh Hydrogen Feed	7800
DMDS (TNPS)	9
TOTAL FEED	319376
STREAMS OUT	
Vent Gas (Dry & N_2 Free)	2357
BTMS Gas (Dry & N_2 Free)	8534
SMT Vent Drain	114
Unit Knockouts	2742
Naphtha Stabilizer Bottoms	58732
ASB	49102
Separated H_2O	47186
RLFVB	143651
TOTAL PRODUCTS	312420
LIQUEFACTION BALANCE %	97.66

TABLE 4.13A
POC-01 SUMMARY OF OPERATING HISTORY

DATE	COND	DESCRIPTION	
10/15/93	Startup	Startup -	Catalyst loaded to K-1, K-2 and K-3.
10/17/93	Startup	Startup -	22,700 kg of Mobil Cat-Cycle oil arrived.
10/21/93	Startup	Startup -	Oil flows started to unit.
10/22/93 - 10/28/93	Startup	Startup -	First and second stage catalyst beds ebullated. The unit was then lined out at 343°C (650°F) and 18.6 MPA (2700 psi) to hydrotreat and distill the startup oil to make a 343°C+ hydrotreated make-up oil.
10/25/93 - 10/28/93	Startup	Startup -	TNPS was injected to presulfide the catalyst.
10/29/93	L/O	Period 1 -	Coal was introduced to the unit at 1300.
10/30/93	L/O	Period 2 -	Line out was completed. Coal feed to the slurry mix tank was stopped for 6.3 hours due to excess dumping of coal into the slurry mix tank when the coal holding tank was filled.
10/31/93	L/O	Period 3 -	Normal operation.
11/1/93	L/O	Period 4 -	Coal feed to slurry mix tank stopped for 1 hour due to excess dumping of coal into the slurry mix tank when the coal holding tank was filled.
11/2/93	L/O Shutdown	Period 5 -	First and second stage reactor temperatures were being increased to 410°C (770°F) and 435°C (815°F) when a high pressure tubing connection on the first stage catalyst withdrawal system failed at 1530 hours. The unit was shutdown in an orderly fashion.
11/7/93	Restart L/O	Period 6 -	Coal feed was resumed at 1500 hours.
11/8/93	L/O	Period 7 -	Normal operation.
11/9/93	L/O	Period 8 -	Several unions in ROSE-SR^{SM} unit were leaking.
11/10/93	L/O	Period 9 -	ROSE-SR^{SM} leaks were being fixed.

TABLE 4.13B
POC-01 SUMMARY OF OPERATING HISTORY

DATE	COND	DESCRIPTION	
11/11/93	L/O Shutdown	Period 10 -	ROSE-SR^{SM} leaks were fixed. A loss of liquid level in the hot separator caused the unit to rapidly lose 6.2 MPa (900 psi) in pressure and carried catalyst from K-2 into the hot separator. The unit was shutdown to inspect and clean out the hot separator and second stage reáctor. The ROSE-SR^{SM} unit was operated successfully during this shutdown.
12/4/93	L/O	Period 11 -	Unit was restarted with coal feed being introduced at 1200 hours. ROSE-SR^{SM} first stage settler top flange leaked and was repaired.
12/5/93	L/O	Period 12 -	Normal operation.
12/6/93	1	Period 13 -	ROSE-SR^{SM} unit brought online.
12/7/93	1	Period 14 -	Normal operation.
12/8/93	1	Period 15 -	At 2200 hours the transfer of slurry from the hot separator to the reactor liquids flash vessel became inhibited and coal feed was stopped.
12/9/93	1	Period 16 -	At 0600 hours the coal feed was restarted to the slurry mix tank. At 1000 hours the hydrotreater was taken offline to obtain untreated distillate samples for monitoring hydrotreater performance.
12/10/93	1	Period 17 -	Hydrotreater brought back online at 1600 hours. At 1445 hours feed to the ROSE-SR^{SM} unit was stopped to allow cleaning of the shell and tube heat exchanger; feed was resumed at 1715 hours.
12/11/93	1	Period 18 -	Normal operation.
12/12/93	1	Period 19 -	Normal operation.
12/13/93	2	Period 20 -	Normal operation.
12/14/93	2	Period 21 -	ROSE-SR^{SM} was offline for 8.5 hours due to a pluggage of the first stage settler bottom outlet..
12/15/93	2	Period 22 -	The unit DP had been rising since Period 19 from 125 psi to 300 psi. Additional water was injected to the hydrotreater outlet and the DP returned to 125 psi.
12/16/93	2	Period 23 -	ROSE-SR^{SM} was offline for 2 hours in order to repair the solvent feed pump.
12/17/93	2	Period 24 -	Normal operation.

TABLE 4.13C
POC-01 SUMMARY OF OPERATING HISTORY

DATE	COND	DESCRIPTION	
12/18/93	2	Period 25 -	The unit DP again rose from 125 psi in Period 22 to 200 psi before returning to the 125 psi range.
12/19/93	2	Period 26 -	Normal operation.
12/20/93	3A	Period 27 -	The ROSE-SR^{SM} unit was taken offline at the start of the period so repairs could be made to the block valves in the transfer line from the first stage settler to the bottoms receiver. ROSE-SR^{SM} was brought back online at 2000 hours. At 0130 hours the fresh feed preheater became jammed causing K-1 and K-2 reactor temperatures to drop to 375°C (708°F) and 427°C (801°F), respectively. This was repaired and reactor temperatures were normal at 0300 hours.
12/21/93	3A	Period 28 -	Normal operation.
12/22/93	3A	Period 29 -	Erratic unit operation caused by level control difficulties in the hot separator, cold separator and scrubber; possible catalyst carryover from K-1 to the hot separator caused by pressure fluctuations. ROSE-SR^{SM} unit taken offline for cleaning.
12/23/93	3A	Period 30 -	Erratic unit operation caused by level control difficulties in the hot separator, cold separator and scrubber.
12/24/93	3A	Period 31 -	Erratic unit operation caused by level control difficulties in the hot separator, cold separator and scrubber.
12/25/93	3A Shutdown	Period 32 -	Erratic unit operation caused by level control difficulties in the hot separator, cold separator and scrubber. Coal feed to unit stopped at 0345 hours after the hot separator letdown system quit passing material.
1/3/94	L/O	Period 33 -	Coal feed was resumed at 0600 hours.
1/4/94	L/O	Period 34 -	Normal operation.
1/5/94	L/O	Period 35 -	At 1435 hours the right side hot separator level control valve trim broke. Over the next 3 hours the hot separator letdown valves jammed.

TABLE 4.13D
POC-01 SUMMARY OF OPERATING HISTORY

DATE	COND	DESCRIPTION	
1/6/94	L/O Shutdown	Period 36 -	At 1823 hours the O-5 level control valve trim broke and caused the unit to rapidly depressure to 2000 psig and carried catalyst over into the hot separator. Coal feed was suspended at 1845 hours and the unit was shutdown.
1/14/94	L/O Shutdown	Period 37 -	Restart of the unit. At the end of the period the hot separator relief valve opened and would not reseat causing rapid depressuring of the unit to 100 psi. Both reactors were flushed and the unit was shutdown.
1/21/94	L/O Shutdown	Period 38 -	Coal feed resumed at 0400 hours. At 2217 hours the ebullating oil flow to the first stage reactor was lost and the unit was shutdown.
1/29/94	L/O	Period 39 -	Coal feed was resumed at 1200 hours.
1/30/94	L/O	Period 40 -	Normal operation.
1/31/94	3B	Period 41 -	Normal operation.
2/1/94	3B	Period 42 -	Normal operation.
2/2/94	3B	Period 43 -	Normal operation.
2/3/94	3B Shutdown	Period 44 -	At 1633 hours ebullation was interrupted in both reactors while catalyst addition was performed to the first stage reactor. Coal feed was stopped. The second stage catalyst addition system became plugged and could not be used for the rest of the run.
2/5/94	L/O	Period 45 -	Coal feed resumed at 0400 hours.
2/6/94	L/O	Period 46 -	First stage catalyst addition line became restricted.
2/7/94	4A	Period 47 -	Normal operation.
2/8/94	4A	Period 48 -	Normal operation.
2/9/94	4B	Period 49 -	Normal operation.
2/10/94	4B	Period 50 -	Coal feed was stopped at the end of the period due to loss of suction by the first stage ebullating pump which caused loss of ebullation.

TABLE 4.13E
POC-01 SUMMARY OF OPERATING HISTORY

DATE	COND	DESCRIPTION
2/11/94	L/O	Period 51 - At 1040 hours ebullation resumed. First stage catalyst addition line was cleared. Coal feed was delayed because our hydrogen supplier could not guarantee delivery due to a heavy winter snow storm. ROSE-SR^{SM} taken offline due to a lack of feed material caused by the coal outage.
2/12/94	L/O	Period 52 - Coal feed resumed at 1700 hours.
2/13/94	L/O	Period 53 - ROSE-SR^{SM} resumed at 1600 hours.
2/14/94	4C	Period 54 - Normal operation.
2/15/94	4C	Period 55 - Normal operation.
2/16/94	4C	Period 56 - Normal operation
2/17/94	4C	Period 57 - Normal operation
2/18/94	5	Period 58 - Special testing of the filter, ebullated bed reactors and alternate hydrotreater feed system were conducted; ROSE-SR^{SM} was operated until the feed ran out at 2150 hours.
2/19/94	Shutdown	Shutdown- Unit was shutdown at 0600 hours.

FIGURE 4.1

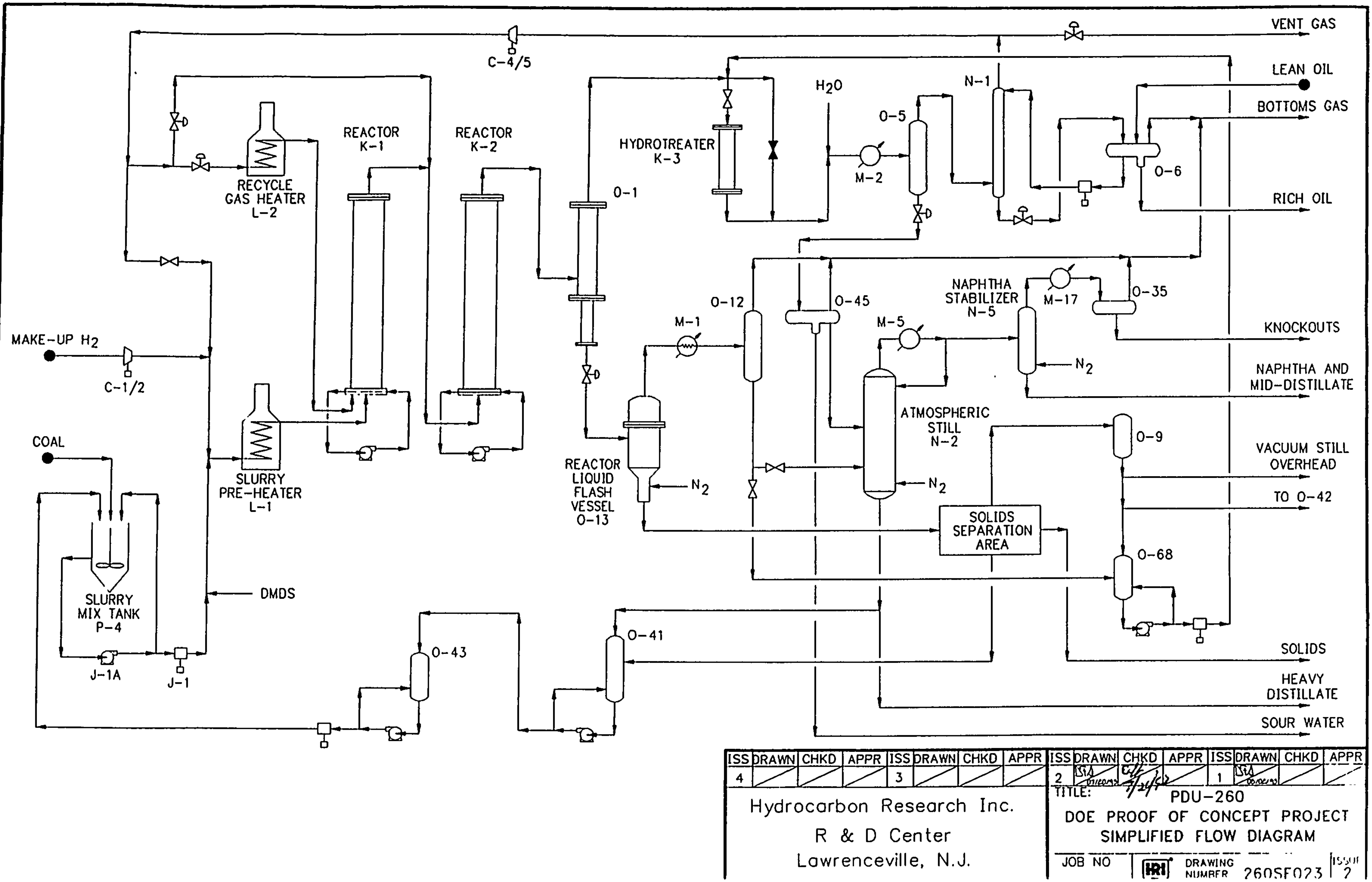
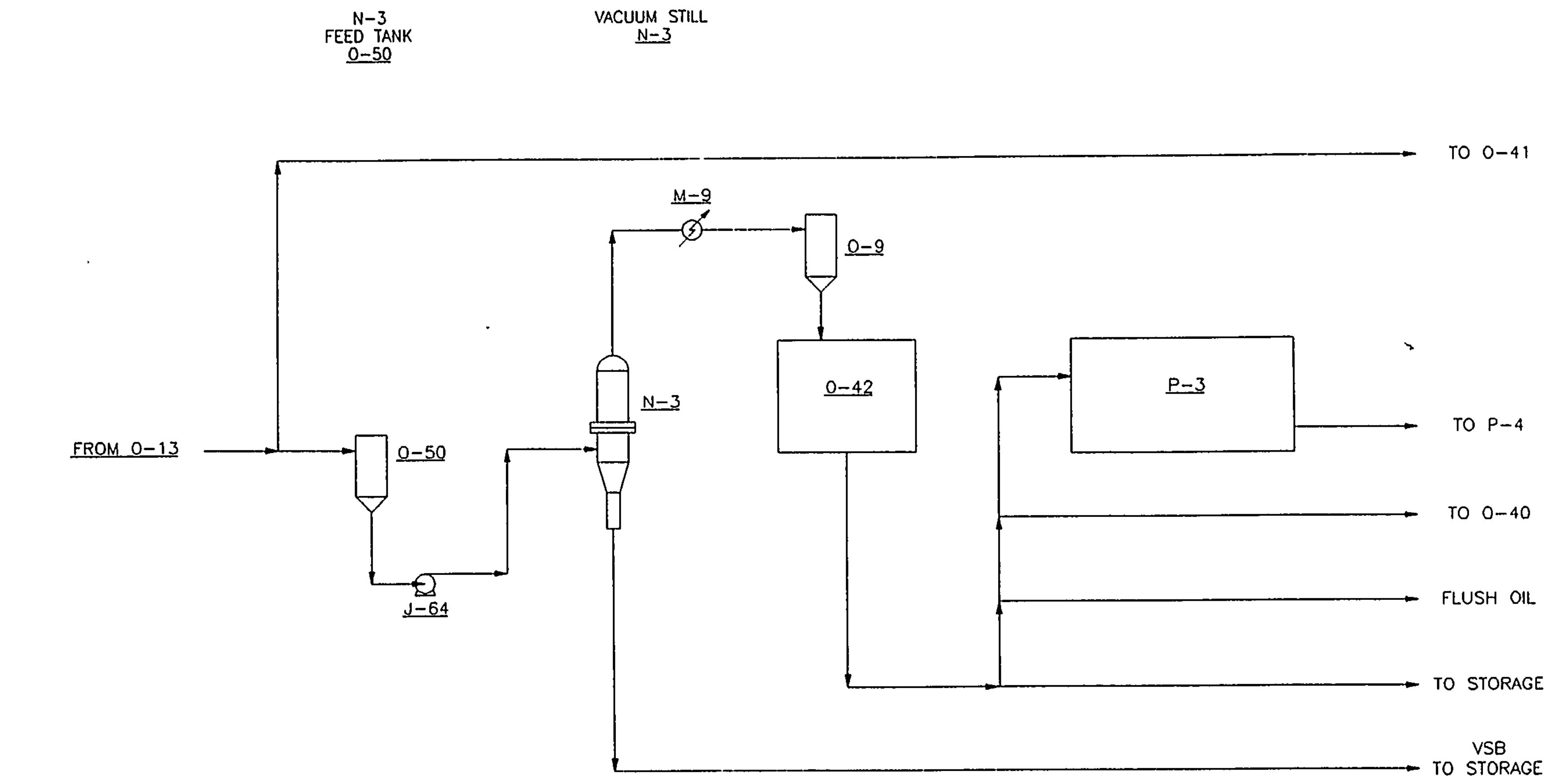

VENT GAS
LEAN OIL
BOTTOMS GAS
RICH OIL
KNOCKOUTS
NAPHTHA AND MID-DISTILLATE
VACUUM STILL OVERHEAD
TO O-42
SOLIDS
HEAVY DISTILLATE
SOUR WATER
MAKE-UP H2
C-1/2
COAL
SLURRY MIX TANK P-4
J-1A
J-1
DMDS
SLURRY PRE-HEATER L-1
RECYCLE GAS HEATER L-2
REACTOR K-1
REACTOR K-2
C-4/5
O-1
HYDROTREATER K-3
H2O
M-2
O-5
N-1
O-6
REACTOR LIQUID FLASH VESSEL O-13
N2
M-1
O-12
O-45
M-5
ATMOSPHERIC STILL N-2
NAPHTHA STABILIZER N-5
M-17
O-35
SOLIDS SEPARATION AREA
O-9
O-68
O-41
O-43
ISS DRAWN CHKD APPR
Hydrocarbon Research Inc.
R & D Center
Lawrenceville, N.J.
TITLE: PDU-260
DOE PROOF OF CONCEPT PROJECT
SIMPLIFIED FLOW DIAGRAM
JOB NO
DRAWING NUMBER 260SF023
ISSUE 2

FIGURE 4.2



ISS	DRAWN	CHKD	APPR	ISS	DRAWN	CHKD	APPR	ISS	DRAWN	CHKD	APPR	ISS	DRAWN	CHKD	APPR
4				3				2				1			

Hydrocarbon Research Inc.
R & D Center
Lawrenceville, N.J.

TITLE:
PDU-260
DOE PROOF OF CONCEPT PROJECT
SIMPLIFIED FLOW DIAGRAM
VACUUM STILL SOLIDS REMOVAL

JOB NO. | HRI | DRAWING NUMBER 260SF025 | ISSUE 1

FIGURE 4.3

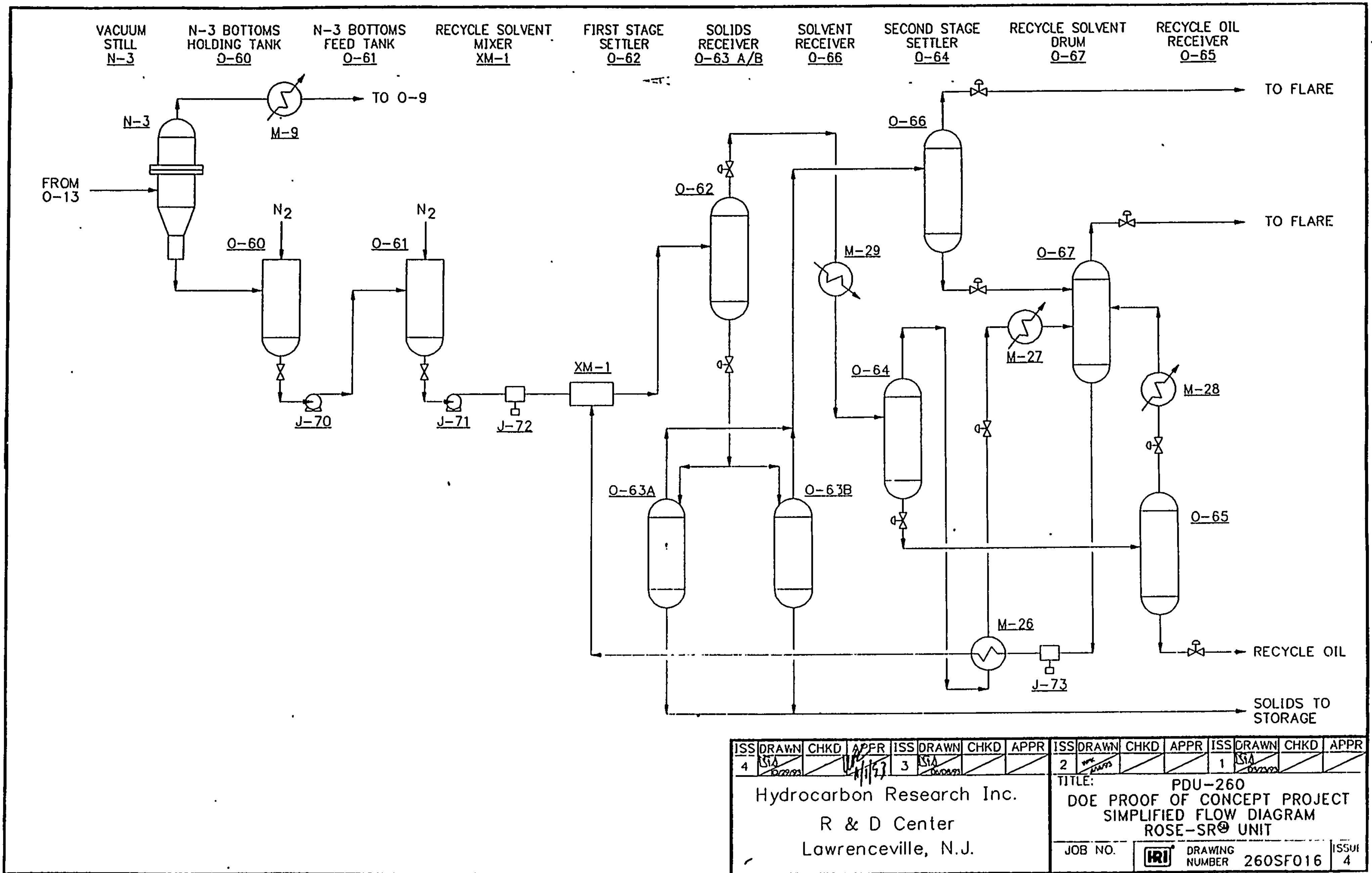
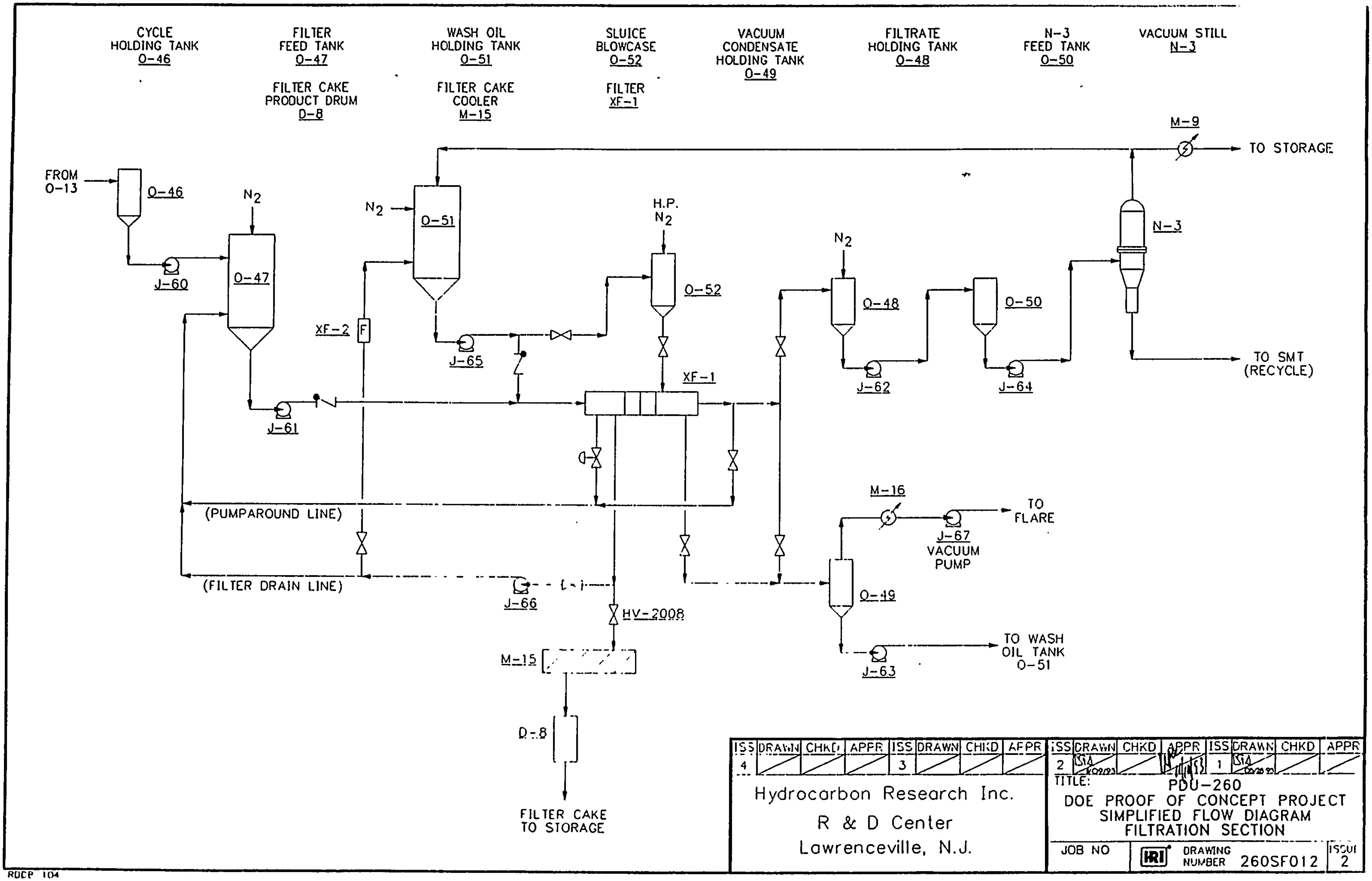

VACUUM STILL N-3
N-3 BOTTOMS HOLDING TANK O-60
N-3 BOTTOMS FEED TANK O-61
RECYCLE SOLVENT MIXER XM-1
FIRST STAGE SETTLER O-62
SOLIDS RECEIVER O-63 A/B
SOLVENT RECEIVER O-66
SECOND STAGE SETTLER O-64
RECYCLE SOLVENT DRUM O-67
RECYCLE OIL RECEIVER O-65
FROM O-13
N-3
M-9
TO O-9
N2
O-60
O-61
J-70
J-71
J-72
XM-1
O-62
O-63A
O-63B
M-29
O-66
O-64
M-27
O-67
M-28
O-65
M-26
J-73
TO FLARE
TO FLARE
RECYCLE OIL
SOLIDS TO STORAGE
ISS DRAWN CHKD APPR ISS DRAWN CHKD APPR ISS DRAWN CHKD APPR ISS DRAWN CHKD APPR
4 3 2 1
Hydrocarbon Research Inc.
R & D Center
Lawrenceville, N.J.
TITLE:
PDU-260
DOE PROOF OF CONCEPT PROJECT
SIMPLIFIED FLOW DIAGRAM
ROSE-SR® UNIT
JOB NO.
HRI
DRAWING NUMBER 260SF016
ISSUE 4
RDDP-104

FIGURE 4.4



CYCLE HOLDING TANK O-46
FILTER FEED TANK O-47
FILTER CAKE PRODUCT DRUM D-8
WASH OIL HOLDING TANK O-51
FILTER CAKE COOLER M-15
SLUICE BLOWCASE O-52
FILTER XF-1
VACUUM CONDENSATE HOLDING TANK O-49
FILTRATE HOLDING TANK O-48
N-3 FEED TANK O-50
VACUUM STILL N-3
FROM O-13
O-46
J-60
N2
O-47
J-61
XF-2
F
N2
O-51
J-65
H.P. N2
O-52
XF-1
N2
O-48
J-62
O-50
J-64
N-3
M-9
TO STORAGE
TO SMT (RECYCLE)
(PUMPAROUND LINE)
(FILTER DRAIN LINE)
J-66
HV-2008
M-15
D-8
FILTER CAKE TO STORAGE
M-16
J-67
VACUUM PUMP
TO FLARE
O-49
J-63
TO WASH OIL TANK O-51
ISS DRAWN CHKD APPR ISS DRAWN CHKD APPR ISS DRAWN CHKD APPR ISS DRAWN CHKD APPR
4
3
2
1
Hydrocarbon Research Inc.
R & D Center
Lawrenceville, N.J.
TITLE:
PDU-260
DOE PROOF OF CONCEPT PROJECT
SIMPLIFIED FLOW DIAGRAM
FILTRATION SECTION
JOB NO
HRI
DRAWING NUMBER 260SF012
ISSUE 2
RDCP 104

FIGURE 4.5

Preparation of Startup and Makeup Oil

L-769: Coal derived gas oil (Tank 4)
L-5669: Light Cycle Oil (Petroleum derived)

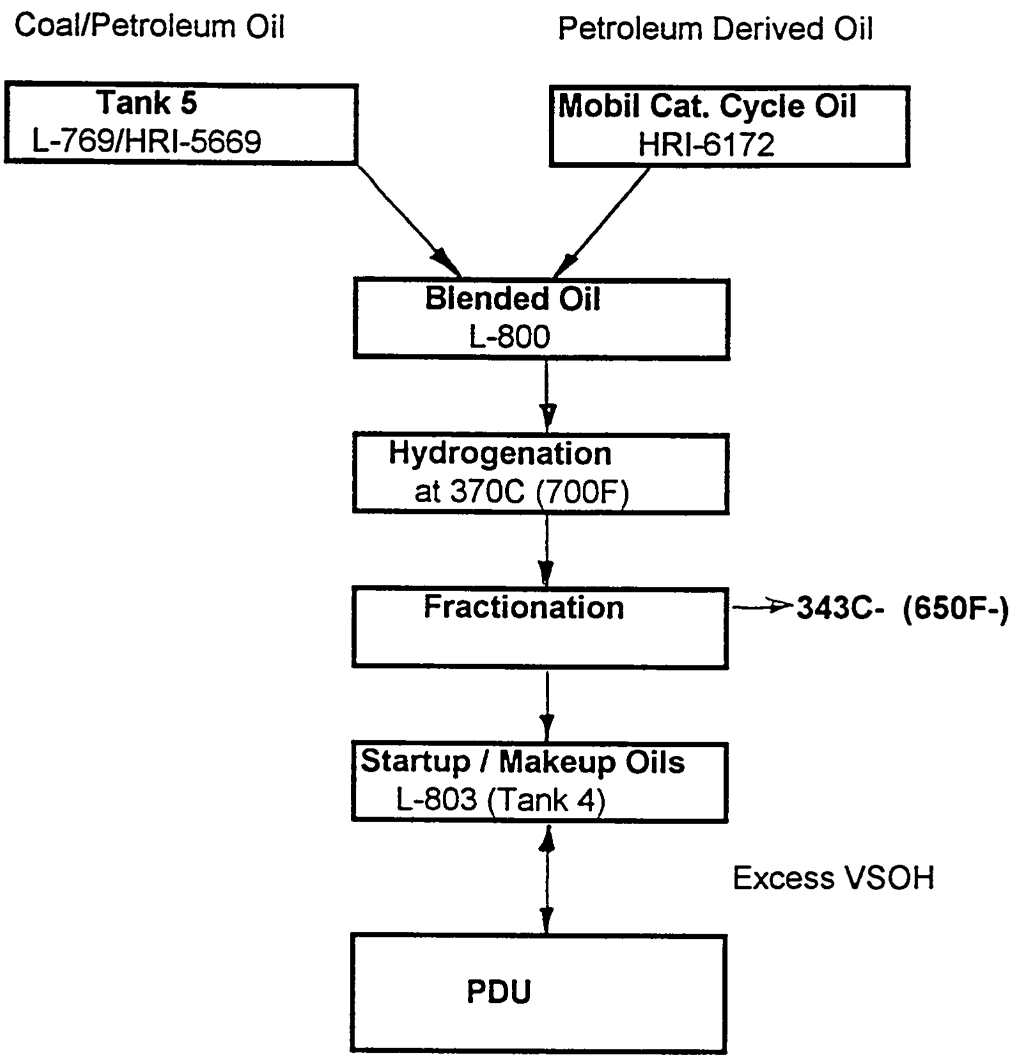


FIGURE 4.6

POC-01 (RUN 260-04) OVERALL MATERIAL BALANCE RECOVERY

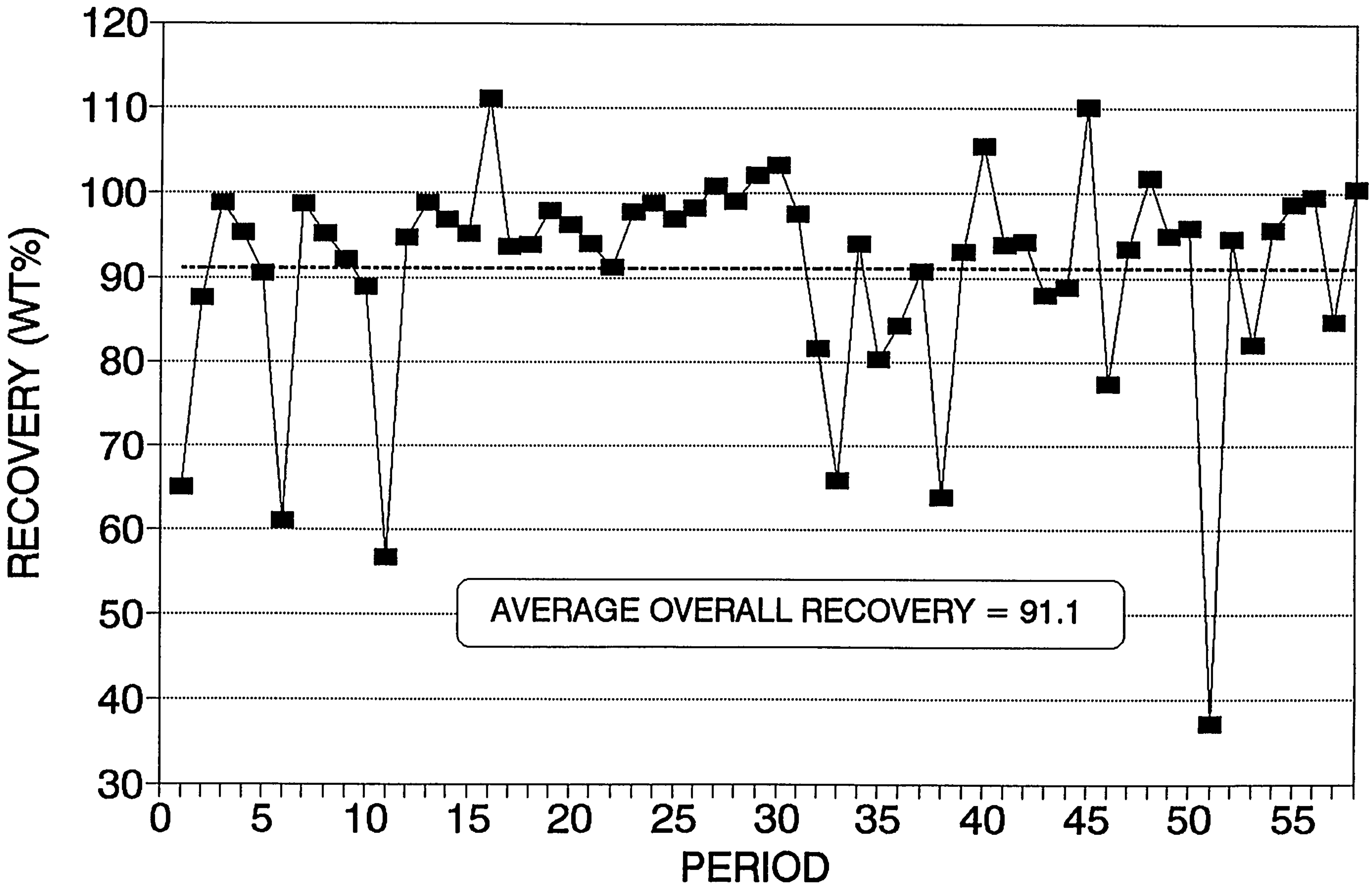


Figure 4.7

POC-01 (RUN 260-04) LIQUEFACTION MATERIAL BALANCE RECOVERY

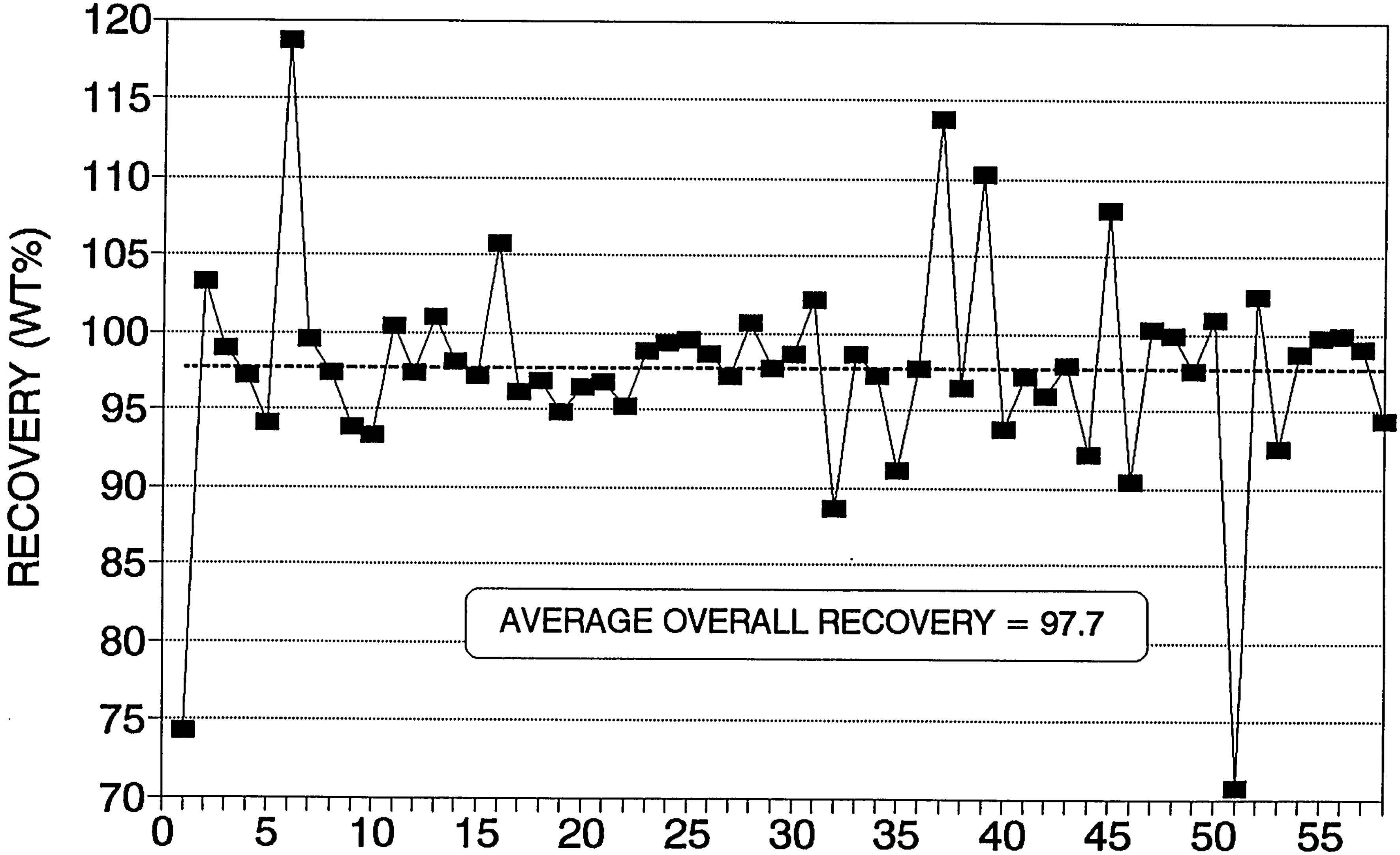


Figure 4.8

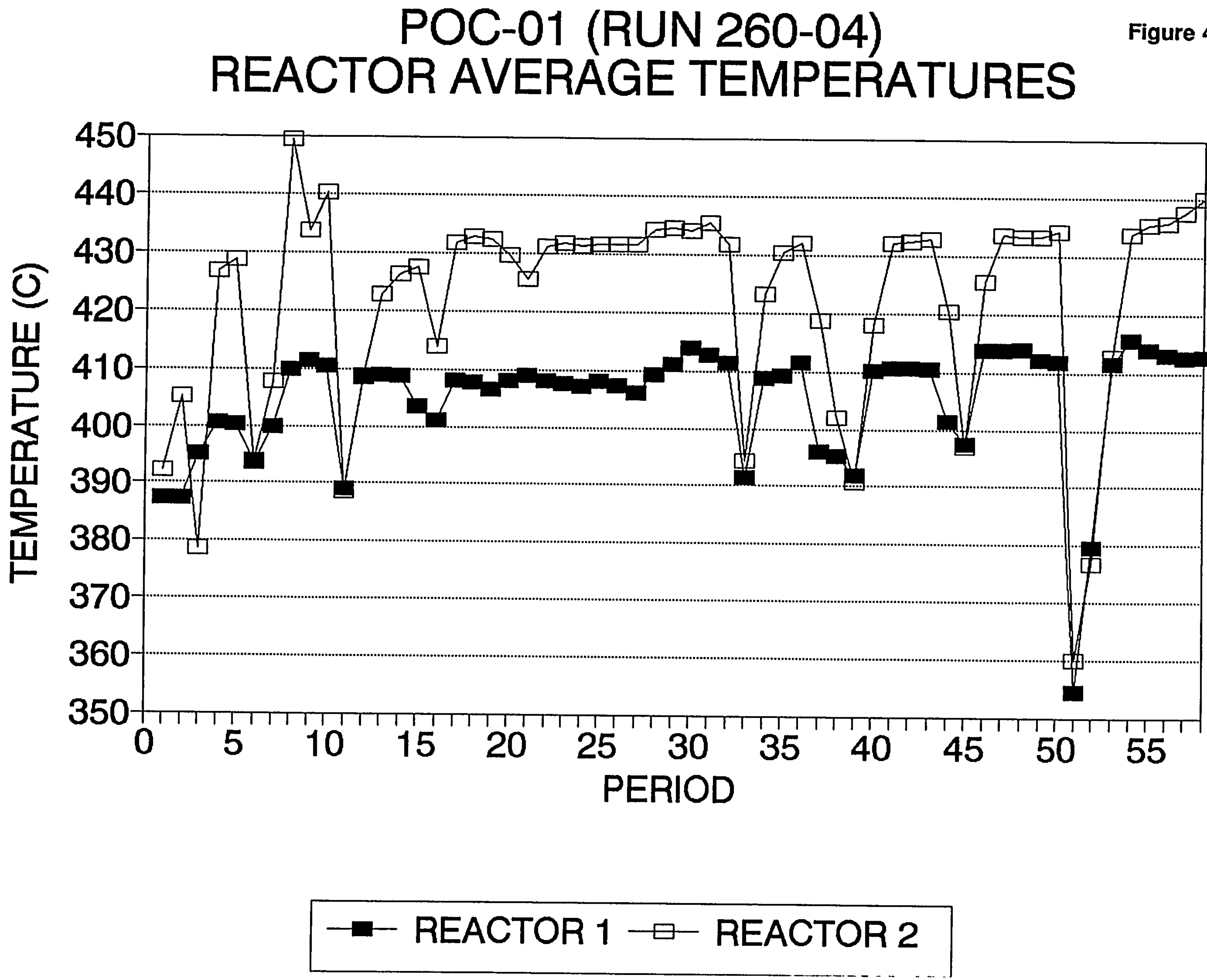

POC-01 (RUN 260-04)
REACTOR AVERAGE TEMPERATURES
TEMPERATURE (C)
450
440
430
420
410
400
390
380
370
360
350
0
5
10
15
20
25
30
35
40
45
50
55
PERIOD
REACTOR 1
REACTOR 2

Figure 4.9

POC-01 (RUN 260-04)
SPACE VELOCITY (DRY COAL)

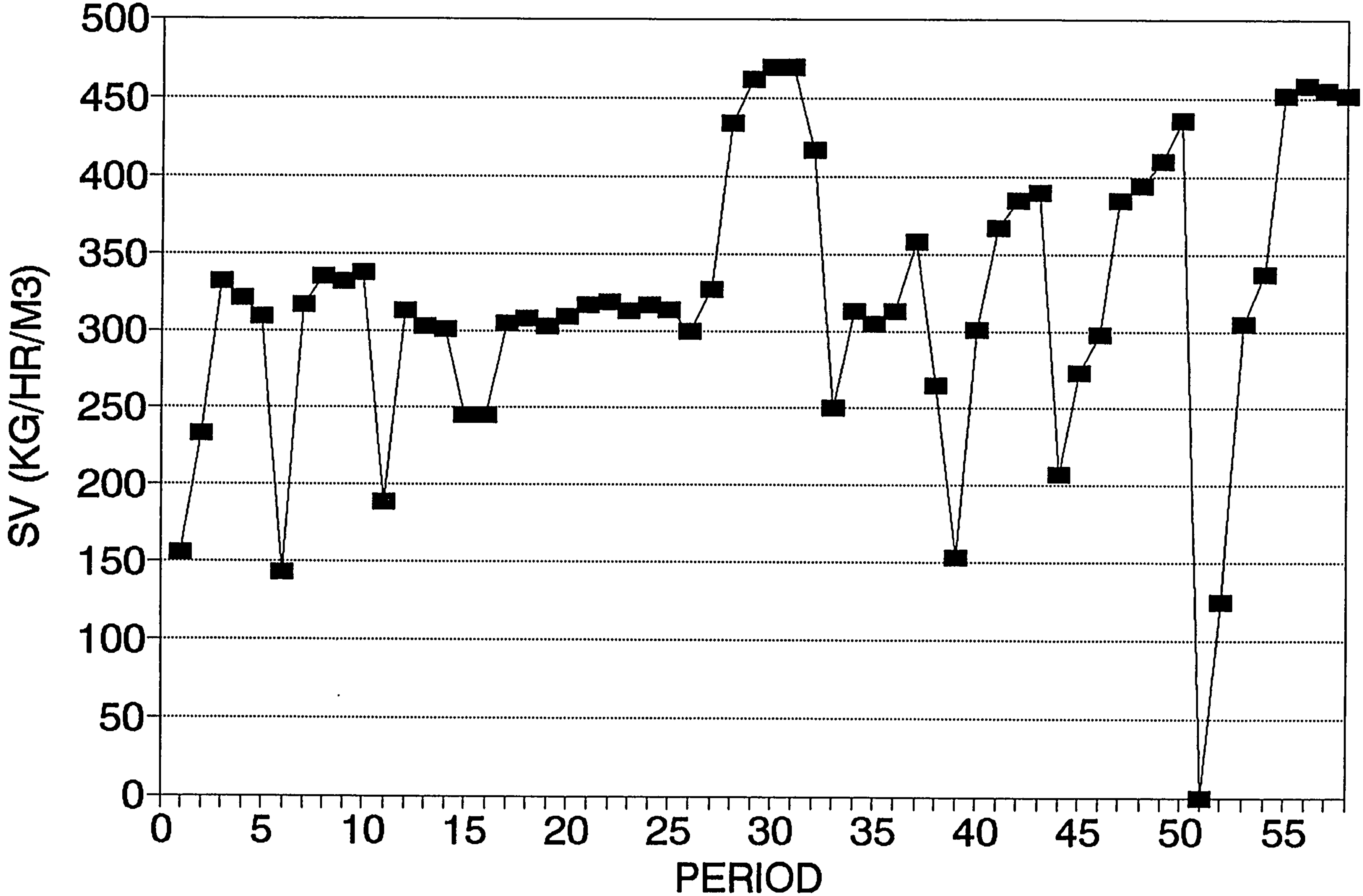


Figure 4.10

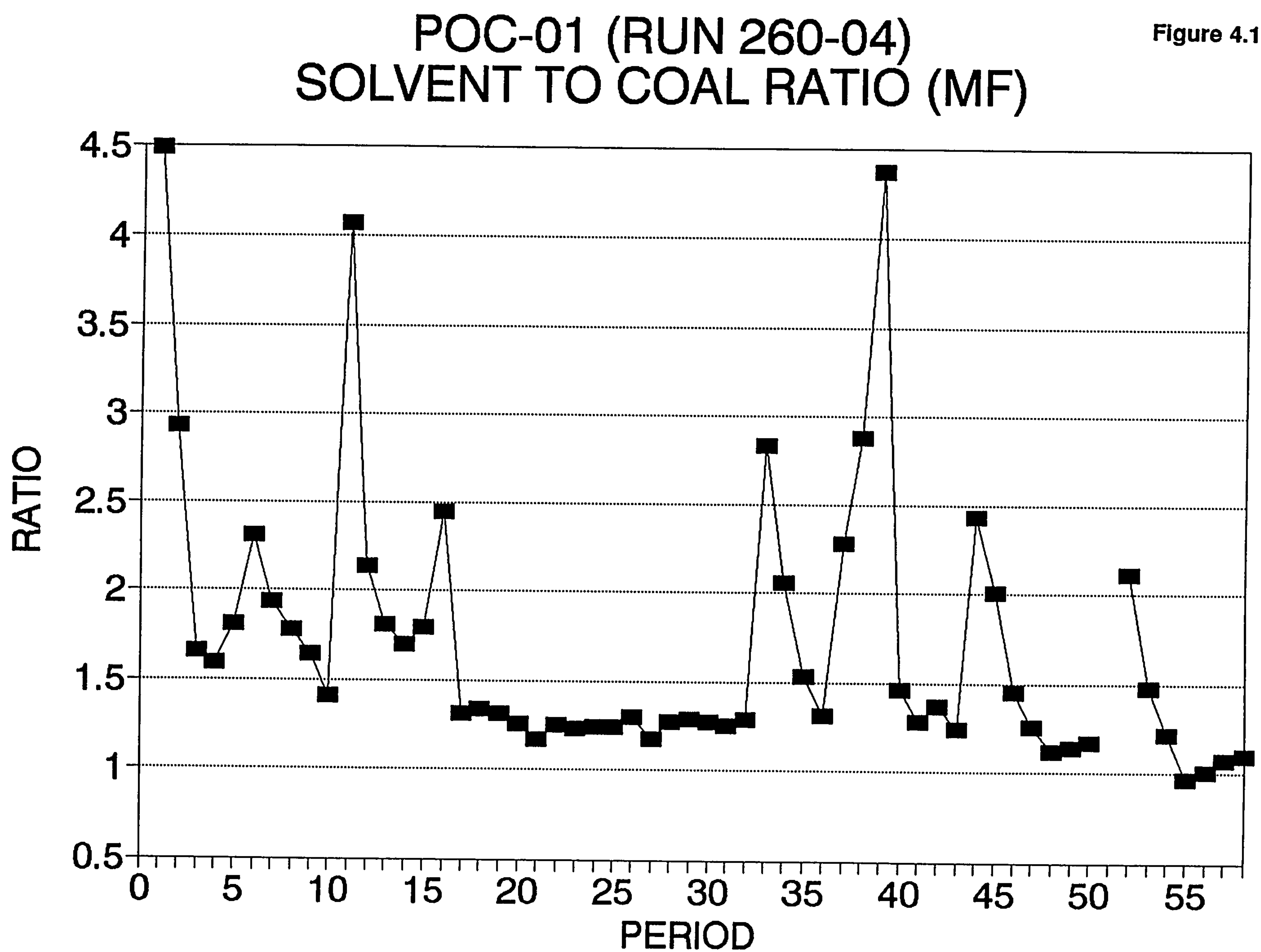

POC-01 (RUN 260-04)
SOLVENT TO COAL RATIO (MF)
RATIO
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
5
10
15
20
25
30
35
40
45
50
55
PERIOD

Figure 4.11

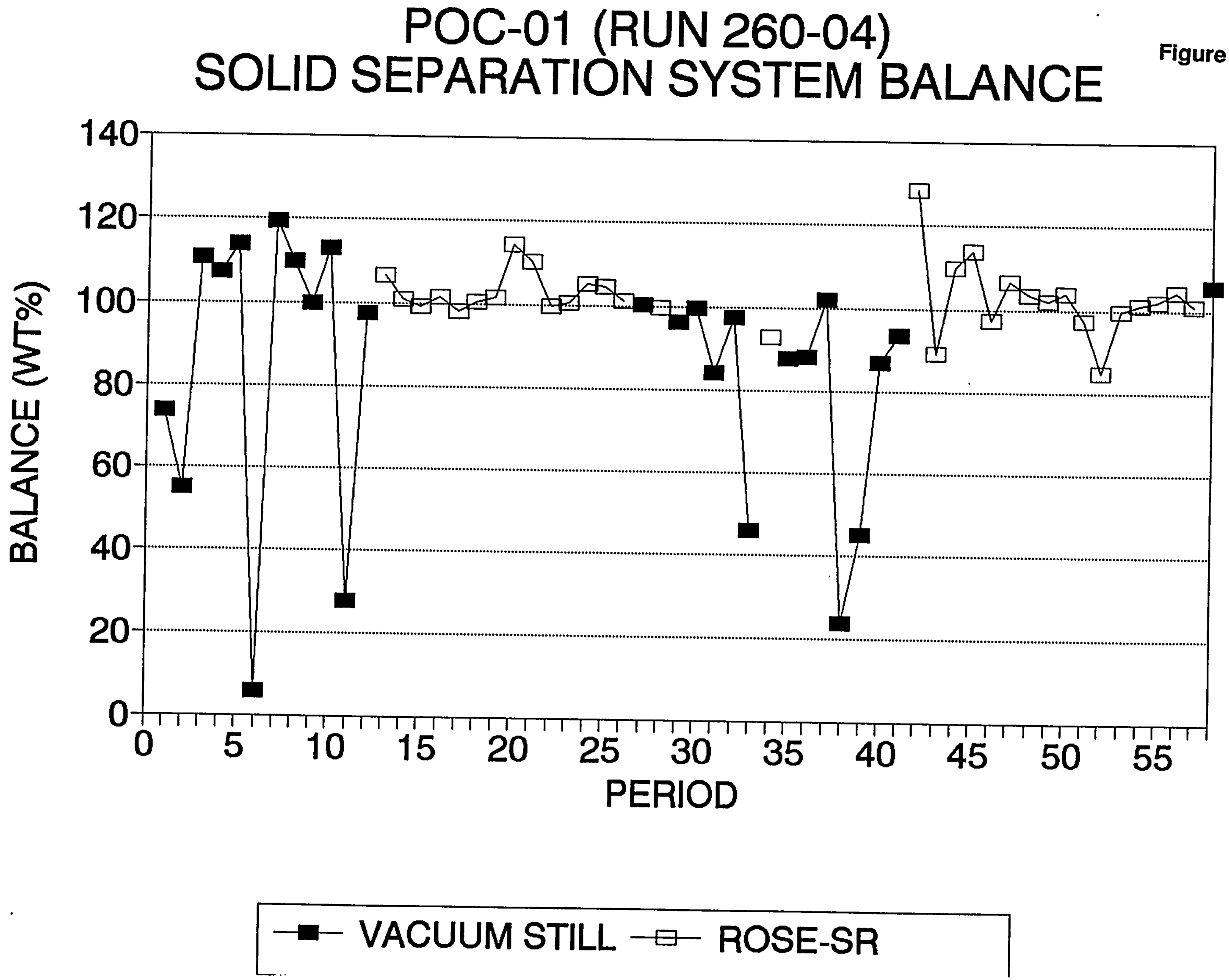
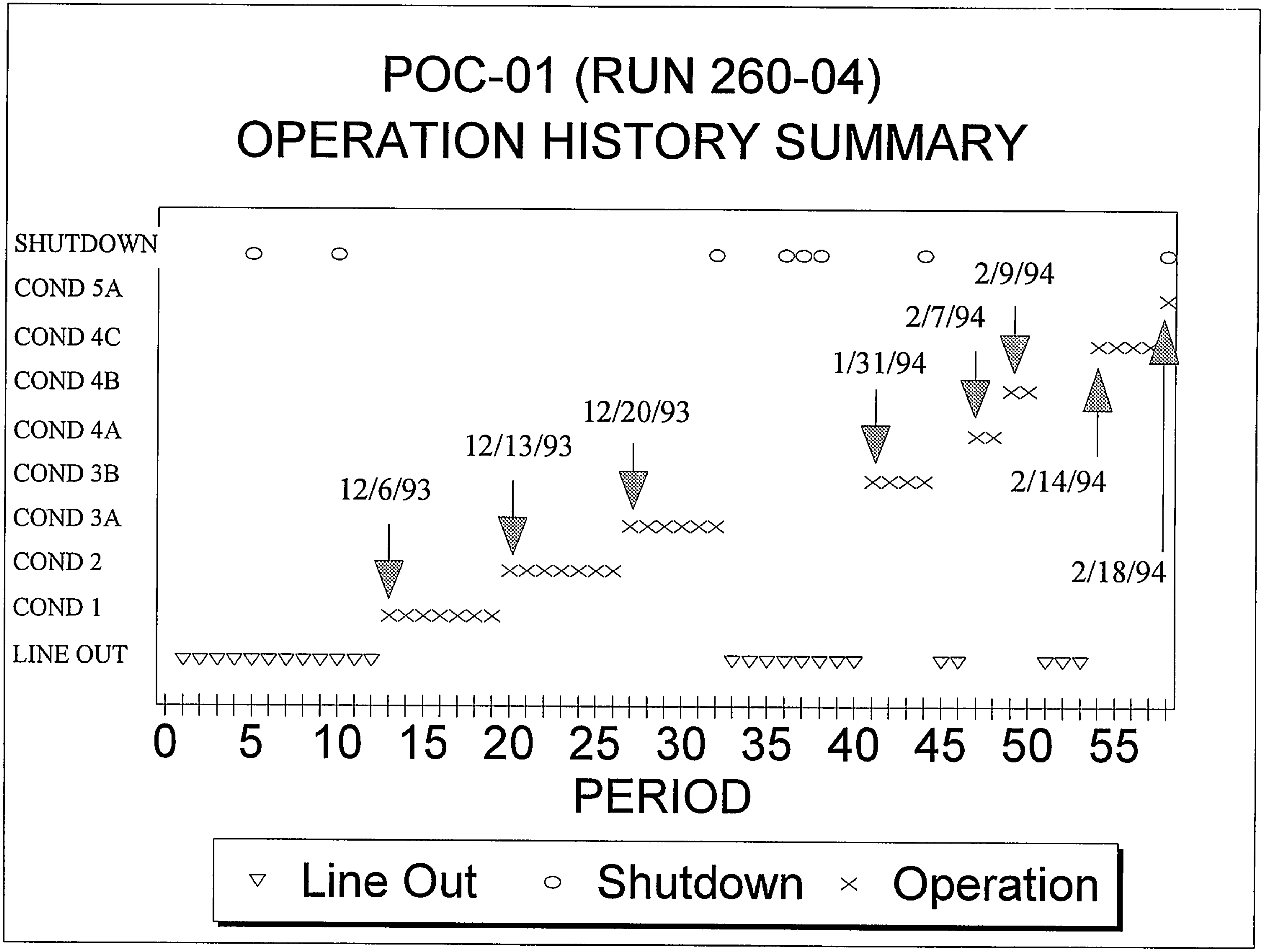

POC-01 (RUN 260-04)
SOLID SEPARATION SYSTEM BALANCE
BALANCE (WT%)
140
120
100
80
60
40
20
0
0
5
10
15
20
25
30
35
40
45
50
55
PERIOD
VACUUM STILL
ROSE-SR

FIGURE 4.12



POC-01 (RUN 260-04)
OPERATION HISTORY SUMMARY
SHUTDOWN
COND 5A
COND 4C
COND 4B
COND 4A
COND 3B
COND 3A
COND 2
COND 1
LINE OUT
12/6/93
12/13/93
12/20/93
1/31/94
2/7/94
2/9/94
2/14/94
2/18/94
0
5
10
15
20
25
30
35
40
45
50
55
PERIOD
Line Out
Shutdown
Operation

SECTION V

PROCESS PERFORMANCE

PDU run POC-01 was a successful scale-up from a 25 kg/day bench scale operation of HRI's CTSL technology. It was a 58 days long run with 8 steady state work-up periods. Process yields were first established, based on the material balance and the analyses of the individual process streams, and normalized based on the Coal Liquefaction Section shown in the Mass Balance Flow Diagram. These yields are elementally balanced, and the ash-balances are also close to 100 Wt%. The reason for normalizing process yields across the Liquefaction Section was the ability to better handle the solids containing stream in the form of reactor flash vessel bottoms (O-13 bottoms) rather than handling streams derived from the solids-separation section. The process performance parameters such as coal and 524°C$^+$ resid conversions, light distillate yields, and heteroatom removals are best represented by the yields across the Liquefaction Section. The performance of the solids-separation and recycle solvent-recovery section has been evaluated separately in terms of the percentage organic and energy rejections, overall coal conversion, and the weight percent rejection of light material (524°C$^-$ distillates) through the bottoms of the solids-separation section. Therefore, in order to get the overall C_4-524°C distillate yield from the process, one has to subtract from the Liquefaction Section distillate yields, the amount of light material rejected with the solids in the bottoms.

A. Process Performance Normalized Yields

As shown in *Table 5.1* and *Figure 5.1* on the POC-01 Process Performance, Illinois No. 6 from Crown II Mine was a very reactive coal, giving over 95 Wt% maf conversions. As high as 75 Wt% maf coal of C_4-524°C distillate yields were obtained. Of these distillates, more than 90 Wt% were liquids boiling below 343°C, as a result of the extinction recycle mode of operations during POC-01 (all the material boiling above 343-398°C was recycled). High levels of organic desulfurization (94-98 Wt%) and denitrogenation (76-88 Wt%) were achieved, even without an on-line hydrotreater unit. Typically, low C_1-C_3 light gas yields (C_1-C_3 selectivities of between 6-12 %) and high hydrogen efficiencies (10-12 %) were also obtained. Figures 5.2 through 5.5 show the process performance during POC-01 PDU run in terms of the light distillate yields vs. process severity and hydrogen consumption, heteroatom removals, and the deasher performance in terms of organic and energy rejections.

For most of Run POC-01, the solids-separation unit consisted of ROSE-SR^{SM} system using n-pentane as the extraction solvent. As indicated in Table 5.1, the on-line operation of the ROSE-SR^{SM} unit was very successful, achieving as low as

12-15 t% organic rejection, 13-17% energy rejection, and absolutely no degradation of the converted coal (maintaining coal conversion value around that based upon the Liquefaction Section). Moreover, the bottoms products from ROSE-SR^{SM} operations were powdery dry in their physical appearance and contained as low as 1-2 Wt% of the light (524°C-) material.

The process mass-balance/yield flow diagrams (with individual stream flow-rates and compositions) for Periods 4, 19, 26, and 57 are indicated in Figures 5.6 through 5.9. As shown in these figures, Period 4 was a line-out condition period with the vacuum still as a deasher (for solids-separation). A lot of make-up oil was used during Period 4, while Periods 19, 26, and 57 utilized small to negligible amounts of make-up oils and were operated with ROSE-SR^{SM} as an on-line solids-separation and recycle solvent-recovery unit. The flow rate of deasher bottoms indicated on all these figures is adjusted for ash-balanced yields. The overall process material recovery for all four representative periods during POC-01, shown in these figures, is also very good (between 97-102 Wt%). It is important to note here that the data presented in Figures 5.6 through 5.9 is also based on the normalized (by mass and elemental balances) stream flow rates, as is the data in the process performance Table 5.1. The only differenece is that the numbers in Table 5.1 are the averaged values for three Periods, chose to represent a particular Run Condition, while those in the Figures 5.6 through 5.9 are the actual numbers representing an individual work-up Period of the Run.

B. Comparison Between POC-01 PDU and CC-16 Bench Run

As mentioned earlier in the catalyst section, a continuous run was carried out in the Bench Unit during the CTSL Program (CC-16) to determine the activity of the new Akzo AO-60 NiMo/alumina catalyst. A comparison has been made between the process performance during the 3 t/d PDU run and performance during the 25 Kg/d Bench Unit to illustrate successful scale-up of operations. Unlike the POC operations, where ROSE-SR^{SM} was used for solids-separation, the bench-scale operations used batch pressure filtrations. Also whereas in the POC operations, the supported catalyst in the reactors was being replaced in a periodic manner, the catalyst in bench-scale operations undergoes a batch-deactivation or aging with time on-stream. Table 5.2 and *Figure 5.10* show the operating conditions and compare the yields and conversions obtained under similar severity conditions during the POC-01 and CC-16 runs. Very similar conversions and distillate yields were obtained in these two runs. POC-01 PDU run performance shows better light distillate yields (C_4-343°C) than Bench run CC-16, because of the extinction recycle mode of operation during the PDU run.

C. Comparison Between POC-01 PDU and Wilsonville Baseline, Improved Baseline (Both Projected), and Run 257-J

An attempt has been made to compare process performance during POC-01 and that reported for Wilsonville PDU Run 257-J and also their original baseline and improved baseline projections of the process performance. The comparisons, made under similar operating/severity conditions, are shown in Tables 5.3 and 5.4, and Figures 5.11 and 5.12. The comparison between POC-01 and Wilsonville baseline projected data indicates similar resid conversions and C_4-454°C distillate yields. The deasher organic rejections are also in the same range. The yields of individual boiling fractions strongly indicate that POC-01 resulted in the production of higher light distillate (C_4-288°C) yields than those projected for the Wilsonville baseline case. A similar yield/conversion pattern is observed when comparing POC-01 with the Wilsonville improved baseline projections (*Table 5.4*). Performance comparison between the actual process yields and conversions, obtained during Wilsonville PDU Run 257, Period J, on Illinois No. 6 coal and under similar operating/severity conditions, shows a much better performance in terms of coal conversions, distillate yields, and deasher organic rejection for POC-01 Periods 42-43 than Wilsonville Run 257-J. Again, as mentioned earlier, POC-01 resulted in higher yields of light distillates than either the projected or the actual Wilsonville yields.

TABLE 5.1
POC-01 Process Performance

Coal : Illinois No. 6 Crown II Mine (10.4 w% Dry Ash)
Catalyst: Akzo AO-60 1/16" NiMo Extrudates in both Reactors

Process Conditions		L/O Rose-SR	1	2	3B	4B	4C
Period/s		14	18-20	24-26	42-43	47-49	55-57
Recycle Type		Ashy	Ashy	Ash-free	Ash-free	Ash-free	Ash-free
Space Velocity, Kg/hr/m3		300.8	306.7	310.4	388	397	455
K-1:	H2 Inlet Pressure, KPa	19214	19230	19267	18800	18897	18793
	Temperature, Deg. C	409	408	407	410.5	414	413
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.17	0.2	0.7	0.7	0**	0**
	Catalyst Age, Kg MF Coal/Kg Cat	315	445	545	818	960	1165
K-2:	H2 Inlet Pressure, KPa	18910	18871	18924	18755	18895	18766
	Temperature, Deg. C	426	432	432	432.5	433	436
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.17	0.4	1.4	1.4	0**	0**
	Catalyst Age, Kg MF Coal/Kg Cat	327	445	493	615	741	1002
Relative Severity Index, STTU*		6.82	8.42	7.83	6.46	6.41	7.20
Flow Rates							
Coal Feed, Kg/hr		68.6	69.5	70.2	87.9	89.8	102.8
Oil Streams to SMT							
	O-43 Recycle to SMT, Kg/hr	97.7	59	53.4	64	61.7	64.5
	Make up Oil, Kg/hr	7.2	9.1	1.4	18.2	17.4	7.4
	VSOH (thru' COT) to SMT, Kg/hr	12	22.4	33.7	32.5	26	32.5
Solvent/Coal Ratio, Kg/Kg		1.7	1.3	1.26	1.3	1.17	1.01
Material & Ash Balances							
Liquefaction Section Recovery, W%		98.1	96	99.1	97	99.2	99.5
Overall Material Recovery, W%		96.9	96	98.1	91.2	96.8	94.5
Normalization Factor		1.02	1.04	1.01	1.03	1.01	1
Ash Balance, W%		103.5	103	104	107.7	103	106
NORMALIZED YIELDS, W% MAF COAL [Based on Liquefaction Section: O-13 Bottoms]							
	H2S	4.34	5.05	3.8	3.68	3.79	3.89
	NH3	1.76	1.48	1.43	1.29	1.17	1.48
	H2O	10.3	9.73	9.79	9.91	10.04	10.32
	COx	0.06	0.26	0.05	0.2	0.14	0.66
	C1-C3	7.3	5.61	5.58	4.51	6.74	7.33
	C4-C6	2.84	2.37	2.35	1.97	3.16	3.03
	IBP-177 C	16.58	15.6	16.45	14.81	14.63	12.1
	177-288 C	19.5	29.29	28.67	26.11	25.83	21.5
	288-343 C	29.62	20.89	17.3	23.3	14.95	13.61
	343-524 C	5.55	3.44	8.5	3.54	3.78	7.73
	524 C+ (Solids-free)	4.76	8.58	8.04	11.25	15.28	15.46
	524 C+ (Tol. Insols)	0.35	0.33	0.31	0.38	1.57	3.7
	Unconverted Coal	4.75	4.38	4.97	5.31	4.92	4.61
PROCESS PERFORMANCE							
Chemical H2-Consumption, W% MAF		7.73	7.37	7.24	6.26	6	5.42
Coal Conversion, W% MAF		95.2	95.6	95.1	94.7	95.1	95.4
524 C+ Conversion, W% MAF		90.1	86.7	86.7	83	78	76
Desulfurization, W%		72.4	86.6	79.3	72.7	73.9	75.8
Denitrogenation, W%		88.2	86	82.5	78.2	75.9	78
C4-343 C Net Distillates, W% MAF		68.5	68.2	64.77	66.19	58.57	50.24
C4-524 C Distillates, W% MAF		74.1	71.9	73.27	69.73	62.35	57.97
C1-C3 Selectivity, Kg/Kg of C4-524 C (X 100)		9.9	7.8	7.6	6.5	10.8	12.6
H2 Efficiency, Kg C4-524 C/Kg H2		9.6	9.8	10.1	11.1	10.4	10.7
DEASHER PERFORMANCE							
Organic Rejection, W% MAF		24.2	22.2	15.2	12.5	21	29.1
Energy Rejection, %		23.1	25.2	16.5	12.8	22.5	33
Deasher Coal Conversion, W% MAF		94.8	95.7	95.1	95.2	95.2	94.9
Deasher Rejection of 524 C- Material, W% MAF		10.4	5.9	2.4	1.5	1.8	3.7

*Severity Index: (1/Space Velocity) X Exp (-E/RT); Where E= 53.8 Kcal/mole and Standard Condition: 399 C @ a Space Velocity of 313 Kg/hr/m3.

** No catalyst addition during Conditions 4B and 4C.

TABLE 5.2
Process Performance Comparison Between Bench Run CC-16 & POC-01

Coal : Illinois No. 6
Catalyst: Akzo AO-60 1/16" NiMo Extrudates in both Reactors

Run		POC-01	POC-01	CC-16
COAL		Crown II Mine	Crown II Mine	B.S. Mine 2
Process		CTSL	CTSL	CTSL
Period/s		18-20	24-26	11-13
Solids-Separation		ROSE-SR	ROSE-SR	FILTER
Recycle Type		Ashy	Ash-free	Ash-free
Space Velocity, Kg/hr/m3 (Stage)		306.7	310.0	300.0
K-1:	Temperature, Deg. C	408.0	407.0	413.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.2	0.7	n/a
	Cat. Age, Kg MF Coal/Kg Cat.	445.0	545.0	235.0
K-2:	Temperature, Deg. C	432.0	432.0	432.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.4	1.4	n/a
	Cat. Age, Kg MF Coal/Kg Cat.	445.0	493.0	235.0
Flow Rates				
Coal Feed, Kg/hr		69.5	70.0	0.7
Solvent/Coal Ratio, Kg/Kg		1.3	1.3	1.3
Material Balances				
Liquefaction Section Recovery, W%		96.0	99.1	n/a
Overall Material Recovery, W%		96.0	98.1	98.0
YIELDS, W% MAF COAL				
[Based on Liquefaction Section]				
	H2S	5.1	3.8	3.2
	NH3	1.5	1.4	1.5
	H2O	9.7	9.8	11.3
	COx	0.3	0.1	0.2
	C1-C3	5.6	5.6	8.9
	C4-177 C	18.0	18.8	24.3
	177-288 C	29.3	28.7	12.2
	288-343 C	20.9	17.3	25.0
	343-524 C	3.4	8.5	11.2
	524 C+	8.9	8.4	3.8
	Unconverted Coal	4.4	5.0	6.3
PROCESS PERFORMANCE				
Chemical H2-Consumption, W% MAF		7.4	7.3	7.8
Coal Conversion, W% MAF		95.6	95.0	93.7
524 C+ Conversion, W% MAF		86.6	86.6	89.8
Desulfurization, W%		86.6	79.3	82.0
Denitrogenation, W%		86.0	82.5	89.0
C4-343 C Net Distillates, W% MAF		68.2	64.8	61.4
C4-524 C Distillates, W% MAF		71.6	73.3	72.6
C1-C3 Selectivity, Kg/Kg of C4-524 C (X 100)		7.8	7.6	12.2
H2 Efficiency, Kg C4-524 C/Kg H2		9.7	10.0	9.3
DEASHER PERFORMANCE				
Organic Rejection, W% MAF		22.2	15.2	17.0
Energy Rejection, %		25.2	16.5	n/a
Deasher Coal Conversion, W% MAF		95.7	95.1	93.7

TABLE 5.3
Process Performance Comparison Between POC-01 & Wilsonville Baseline Data (I)

Coal : Illinois No. 6

Run		POC-01	POC-01	Wilsonville
COAL		Crown II Mine	Crown II Mine	B.S. Mine 2
Ash %		10.4	10.4	11.2
Catalyst		AO-60; 1/16"	AO-60; 1/16"	Amocat 1C; 1/12"
Period/s		18-20	24-26	Baseline (Projection)
Solids-Separation		ROSE-SR	ROSE-SR	ROSE-SR
Recycle Type		Ashy	Ash-free	Ashy
Space Velocity, Kg/hr/m3 (Stage)		306.7	310.0	n/a
K-1:	Temperature, Deg. C	408.0	407.0	421.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.2	0.7	1.4
	Cat. Age, Kg MF Coal/Kg Cat.	445.0	545.0	n/a
K-2:	Temperature, Deg. C	432.0	432.0	404.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.4	1.4	0.7
	Cat. Age, Kg MF Coal/Kg Cat.	445.0	493.0	n/a
Flow Rates				
Coal Feed, Kg/hr		69.5	70.0	n/a
Solvent/Coal Ratio, Kg/Kg		1.3	1.3	2.5
Material Balances				
Liquefaction Section Recovery, W%		96.0	99.1	n/a
Overall Material Recovery, W%		96.0	98.1	n/a
YIELDS, W% MAF COAL [Based on Liquefaction Section]				
	H2S+H2O+NH3+COx	16.6	15.1	14.0
	C1-C3	5.6	5.6	4.8
	C4-177 C	18.0	18.8	16.9
	177-288 C	29.3	28.7	19.2
	288-454 C	22.1	19.3	35.1
	454 C+ (+ unconverted coal)	19.3	20.3	16.2
PROCESS PERFORMANCE				
Chemical H2-Consumption, W% MAF		7.4	7.3	6.2
Coal Conversion, W% MAF		95.6	95.0	92.8
454 C+ Resid Conversion, W% MAF		80.7	79.7	83.8
Desulfurization, W%		86.6	79.3	n/a
Denitrogenation, W%		86.0	82.5	n/a
C4-454 C Net Distillates, W% MAF		69.4	67.3	71.2
Deasher Organic Rejection, W% MAF		22.2	15.2	16.3

TABLE 5.4
Process Performance Comparison Between POC-01 & Wilsonville Baseline Data (II)

Coal : Illinois No. 6

<u>Run</u>		POC-01	POC-01	Wilsonville (Projection)	Wilsonville 257
COAL		Crown II Mine	Crown II Mine	B.S. Mine 2	B.S. Mine 2
Ash %		10.4	10.4	11.2	11.2
Catalyst		AO-60; 1/16"	AO-60; 1/16"	Amocat 1C; 1/12"	Amocat 1C; 1/12"
Period/s		42-43	47-49	Improved Baseline	257 J
Solids-Separation		ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
Recycle Type		Ash-free	Ash-free	Ashy	Ashy
Space Velocity, Kg/hr/m3 (Stage)		388.0	397.0	443.0	495.0
K-1:	Temperature, Deg. C	410.0	414.0	432.0	432.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.7	0.0	1.4	1.4
	Cat. Age, Kg MF Coal/Kg Cat.	818.0	960.0	n/a	644.0
K-2:	Temperature, Deg. C	432.0	432.0	404.0	404.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	1.4	0.0	0.7	0.7
	Cat. Age, Kg MF Coal/Kg Cat.	615.0	741.0	n/a	1309.0
<u>Flow Rates</u>					
Coal Feed, Kg/hr		88.0	90.0	n/a	106.0
Solvent/Coal Ratio, Kg/Kg		1.3	1.3	2.3	2.3
<u>Material Balances</u>					
Liquefaction Section Recovery, W%		97.0	99.1	n/a	n/a
Overall Material Recovery, W%		91.0	97.0	n/a	n/a
<u>YIELDS, W% MAF COAL</u> [Based on Liquefaction Section]					
	H2S+H2O+NH3+COx	15.1	15.1	13.9	15.1
	C1-C3	4.5	6.7	5.5	5.4
	C4-177 C	16.8	17.8	15.8	14.5
	177-288 C	26.1	25.8	19.3	18.2
	288-454 C	25.3	17.2	36.1	33.2
	454 C+ (+ unconverted coal)	18.7	23.8	15.7	19.7
<u>PROCESS PERFORMANCE</u>					
Chemical H2-Consumption, W% MAF		6.3	6.0	6.3	6.0
Coal Conversion, W% MAF		94.7	95.1	92.9	91.7
454 C+ Resid Conversion, W% MAF		81.3	76.2	84.3	80.3
Desulfurization, W%		73.0	74.0	n/a	n/a
Denitrogenation, W%		78.0	75.9	n/a	n/a
C4-454 C Net Distillates, W% MAF		68.2	60.8	71.2	65.8
Deasher Organic Rejection, W% MAF		12.5	21.0	15.7	19.0

FIGURE 5.1

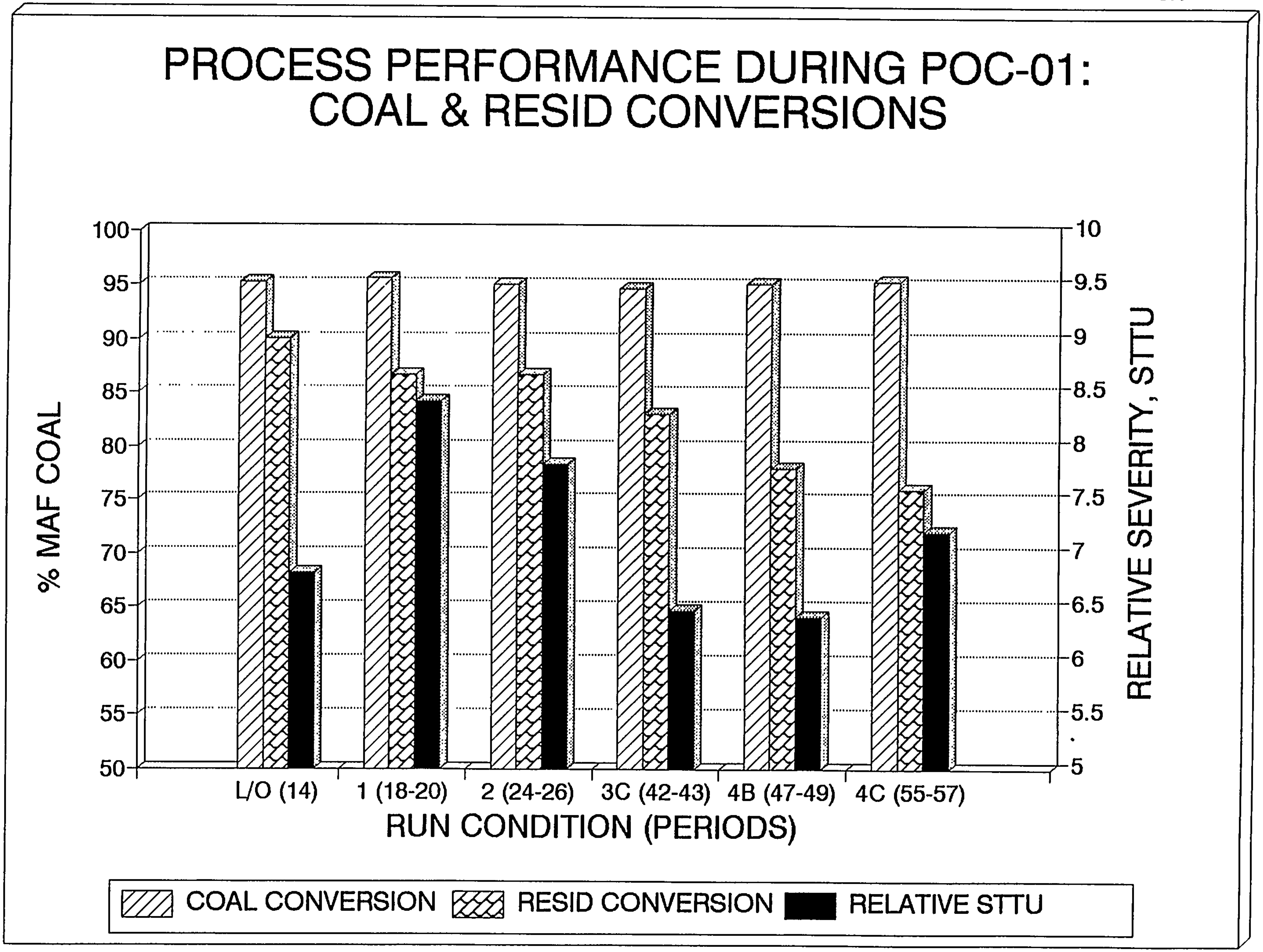

PROCESS PERFORMANCE DURING POC-01:
COAL & RESID CONVERSIONS
% MAF COAL
100
95
90
85
80
75
70
65
60
55
50
RELATIVE SEVERITY, STTU
10
9.5
9
8.5
8
7.5
7
6.5
6
5.5
5
L/O (14)
1 (18-20)
2 (24-26)
3C (42-43)
4B (47-49)
4C (55-57)
RUN CONDITION (PERIODS)
COAL CONVERSION
RESID CONVERSION
RELATIVE STTU

FIGURE 5.2

PROCESS PERFORMANCE DURING POC-01: LIGHT DISTILLATE YIELDS

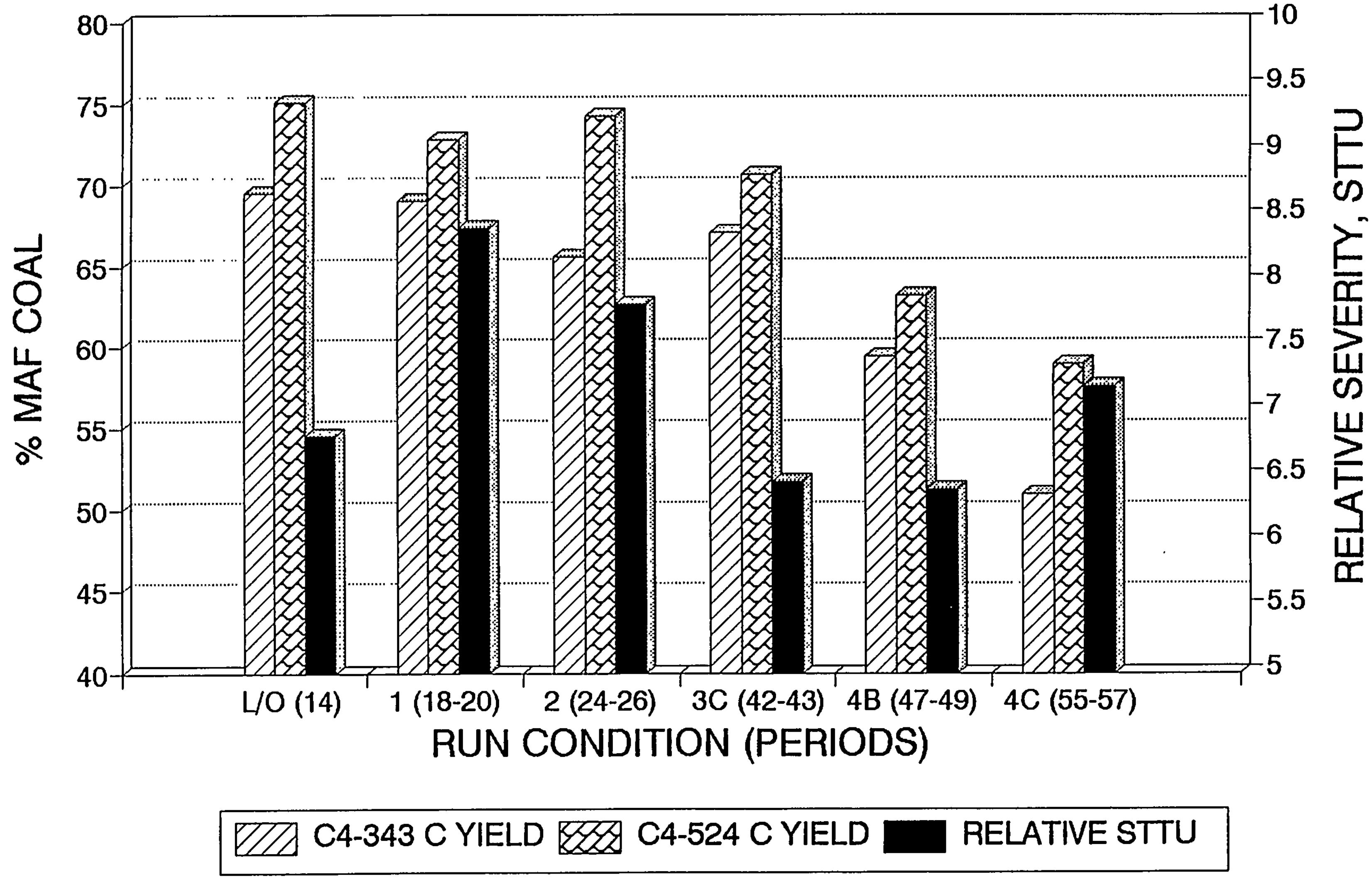


FIGURE 5.3

PROCESS PERFORMANCE DURING POC-01: LIGHT DISTILLATE YIELDS

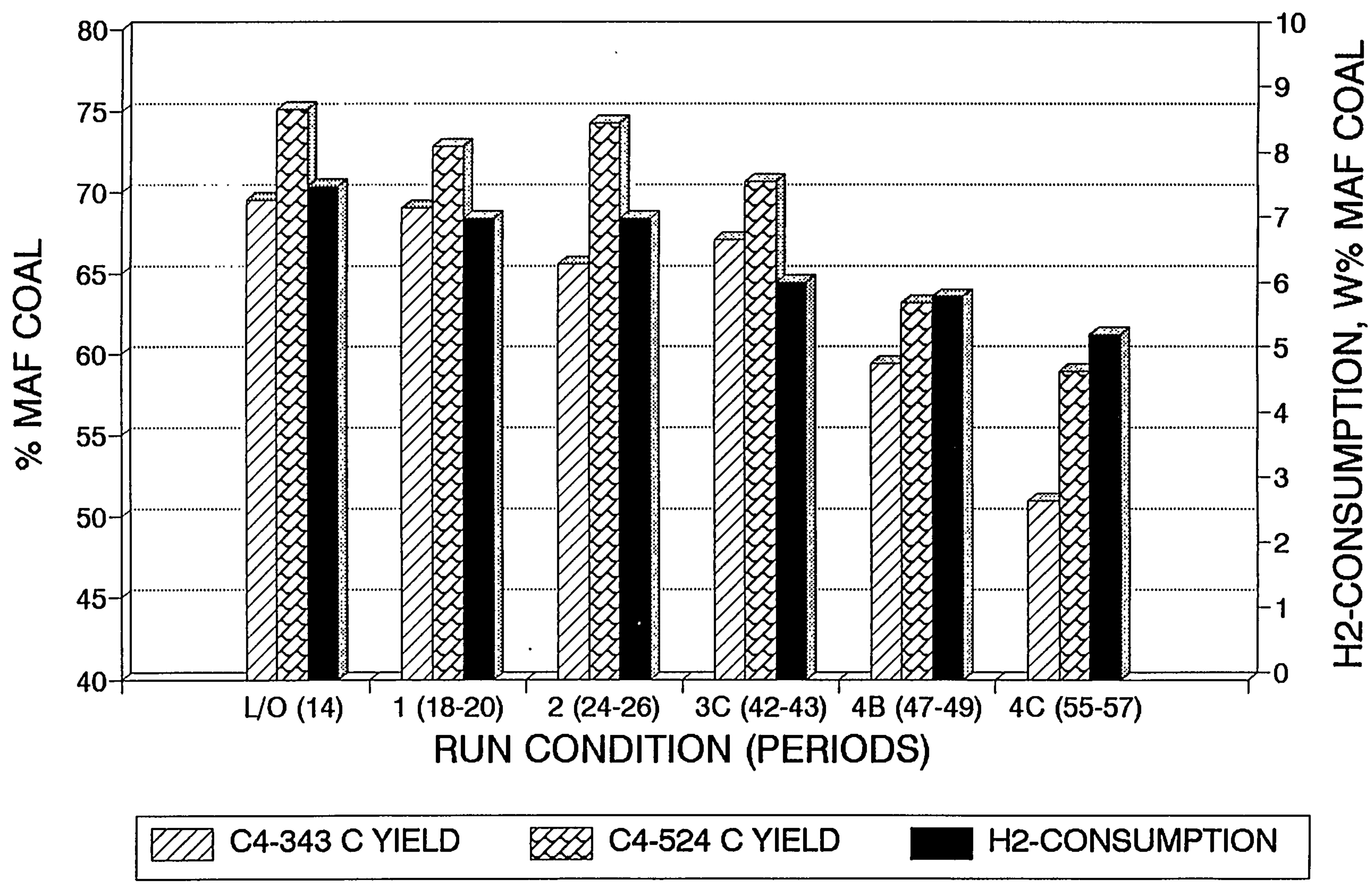
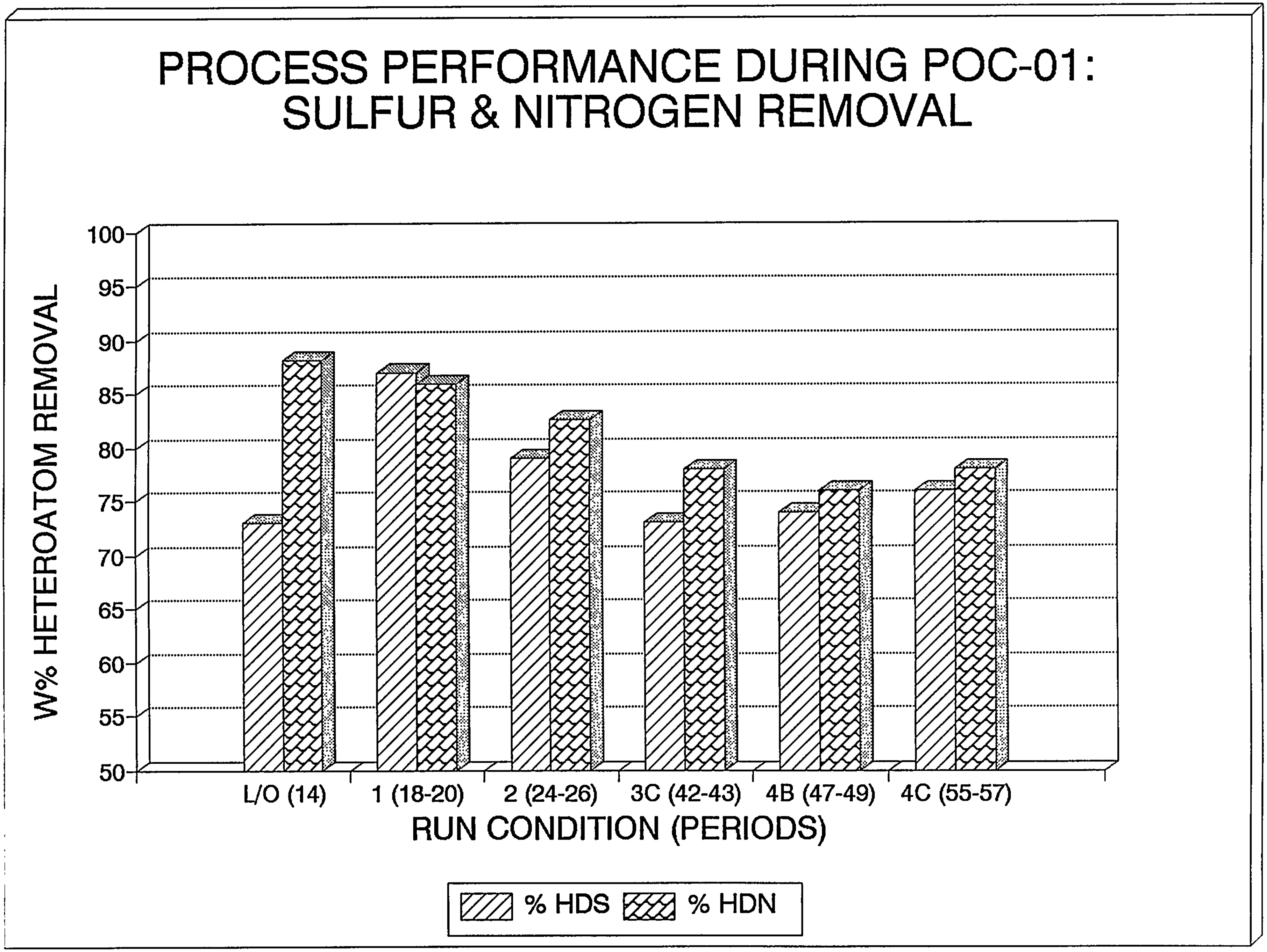


FIGURE 5.4



PROCESS PERFORMANCE DURING POC-01:
SULFUR & NITROGEN REMOVAL
W% HETEROATOM REMOVAL
100
95
90
85
80
75
70
65
60
55
50
L/O (14)
1 (18-20)
2 (24-26)
3C (42-43)
4B (47-49)
4C (55-57)
RUN CONDITION (PERIODS)
% HDS
% HDN

FIGURE 5.5

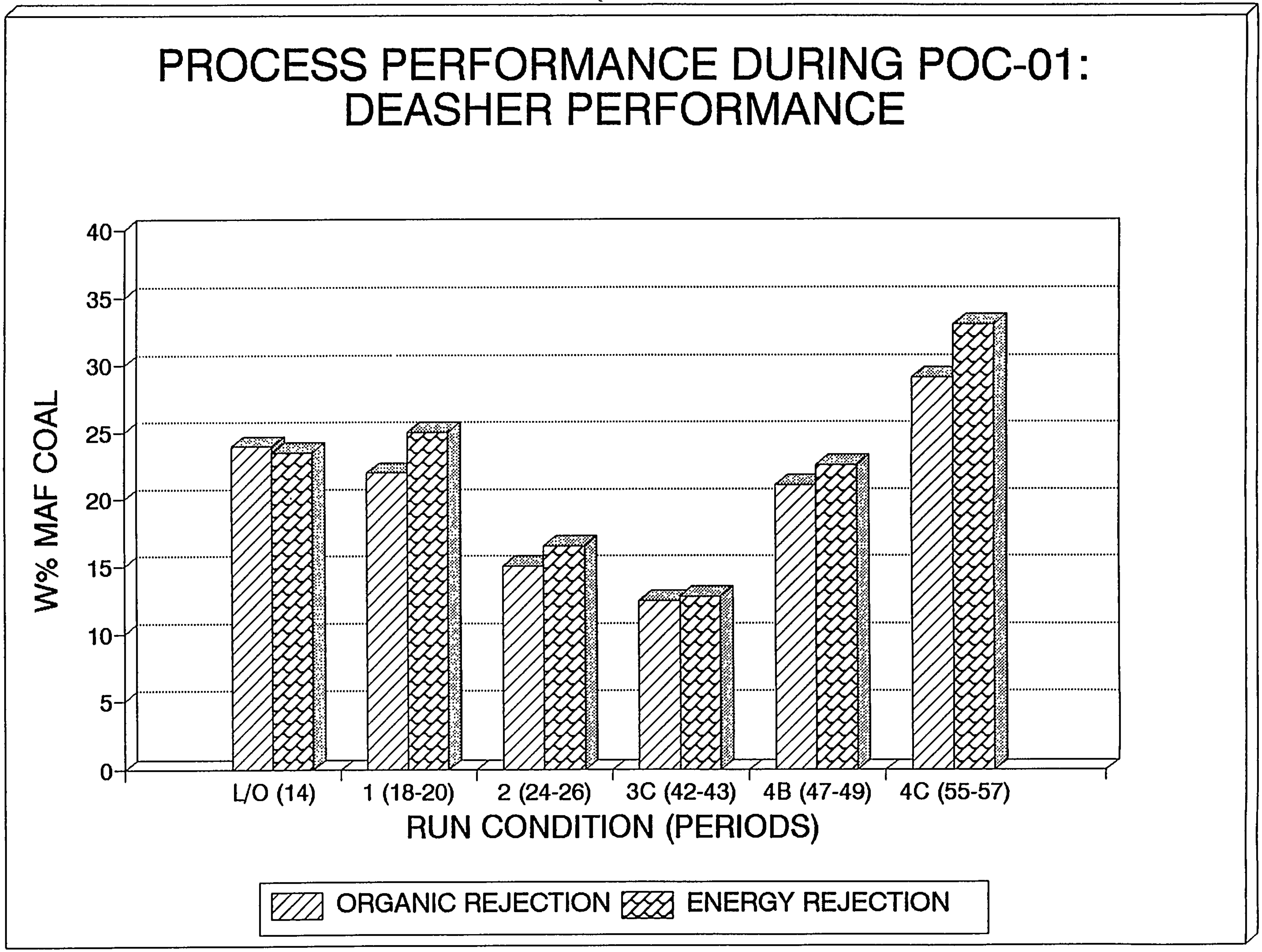

PROCESS PERFORMANCE DURING POC-01:
DEASHER PERFORMANCE
W% MAF COAL
40
35
30
25
20
15
10
5
0
L/O (14)
1 (18-20)
2 (24-26)
3C (42-43)
4B (47-49)
4C (55-57)
RUN CONDITION (PERIODS)
ORGANIC REJECTION
ENERGY REJECTION

FIGURE 5.6

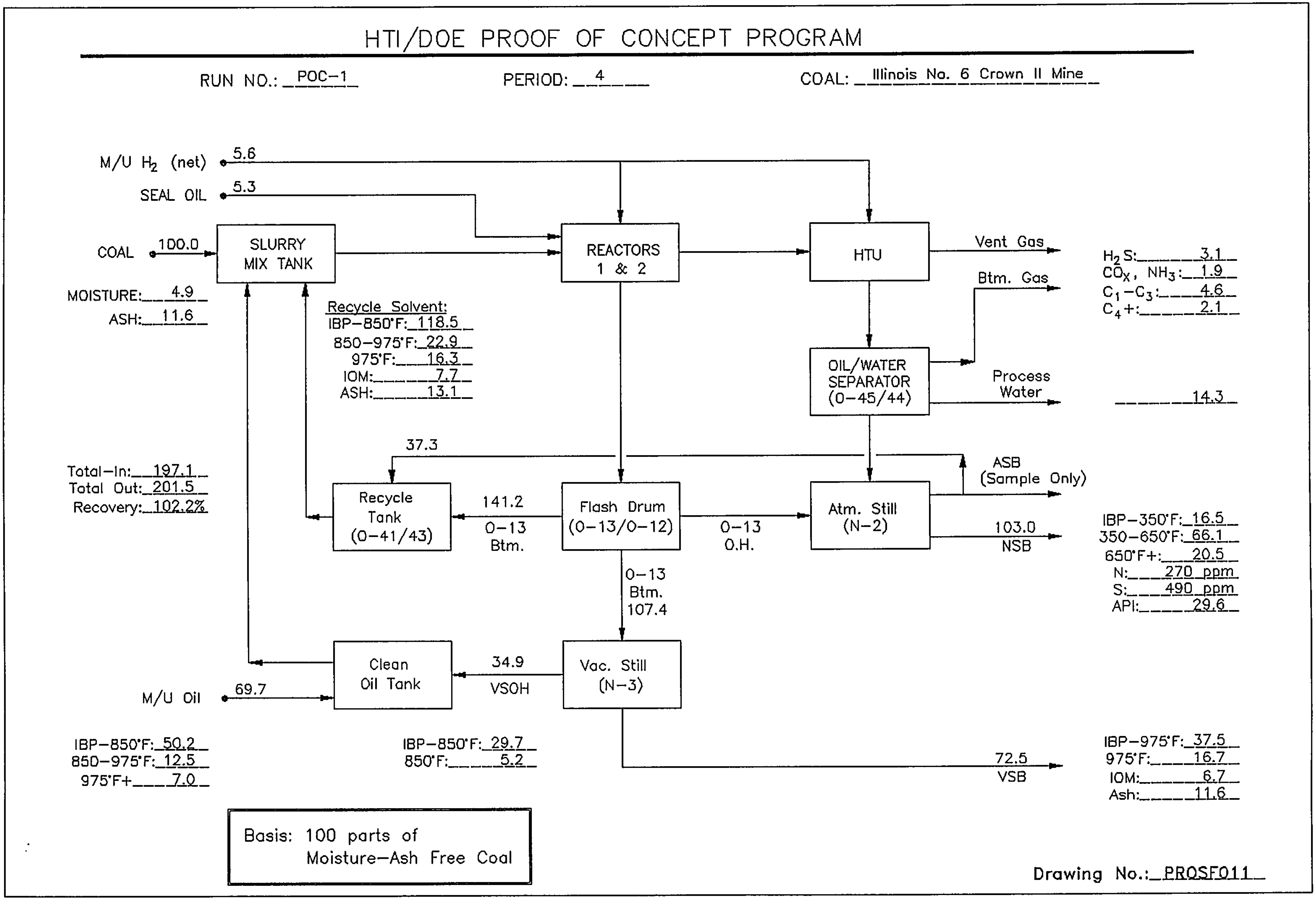
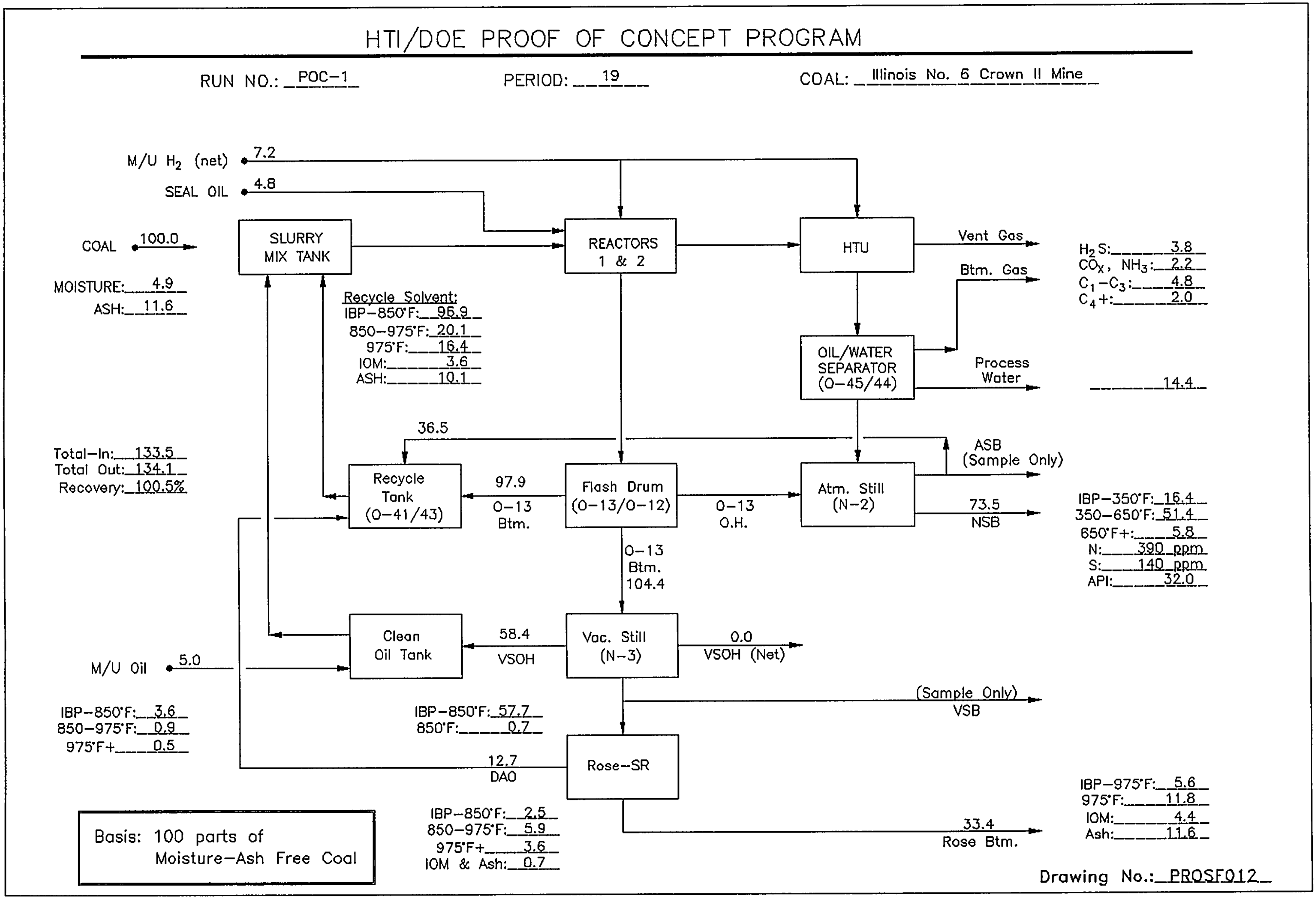

HTI/DOE PROOF OF CONCEPT PROGRAM
RUN NO.: POC-1
PERIOD: 4
COAL: Illinois No. 6 Crown II Mine
M/U H2 (net) 5.6
SEAL OIL 5.3
COAL 100.0
MOISTURE: 4.9
ASH: 11.6
SLURRY MIX TANK
REACTORS 1 & 2
HTU
Vent Gas
H2S: 3.1
COx, NH3: 1.9
C1-C3: 4.6
C4+: 2.1
Btm. Gas
OIL/WATER SEPARATOR (0-45/44)
Process Water
14.3
Recycle Solvent:
IBP-850°F: 118.5
850-975°F: 22.9
975°F: 16.3
IOM: 7.7
ASH: 13.1
37.3
ASB (Sample Only)
Total-In: 197.1
Total Out: 201.5
Recovery: 102.2%
Recycle Tank (0-41/43)
141.2
0-13 Btm.
Flash Drum (0-13/0-12)
0-13 O.H.
Atm. Still (N-2)
103.0
NSB
IBP-350°F: 16.5
350-650°F: 66.1
650°F+: 20.5
N: 270 ppm
S: 490 ppm
API: 29.6
0-13 Btm. 107.4
Clean Oil Tank
34.9
VSOH
Vac. Still (N-3)
M/U Oil 69.7
IBP-850°F: 50.2
850-975°F: 12.5
975°F+ 7.0
IBP-850°F: 29.7
850°F: 5.2
72.5
VSB
IBP-975°F: 37.5
975°F: 16.7
IOM: 6.7
Ash: 11.6
Basis: 100 parts of Moisture-Ash Free Coal
Drawing No.: PROSF011

FIGURE 5.7



HTI/DOE PROOF OF CONCEPT PROGRAM
RUN NO.: POC-1
PERIOD: 19
COAL: Illinois No. 6 Crown II Mine
M/U H_2 (net) 7.2
SEAL OIL 4.8
COAL 100.0
MOISTURE: 4.9
ASH: 11.6
SLURRY MIX TANK
REACTORS 1 & 2
HTU
Vent Gas
Btm. Gas
H_2S: 3.8
CO_x, NH_3: 2.2
C_1-C_3: 4.8
C_4+: 2.0
Recycle Solvent:
IBP-850°F: 95.9
850-975°F: 20.1
975°F: 16.4
IOM: 3.6
ASH: 10.1
OIL/WATER SEPARATOR (O-45/44)
Process Water
14.4
36.5
ASB (Sample Only)
Total-In: 133.5
Total Out: 134.1
Recovery: 100.5%
Recycle Tank (O-41/43)
97.9
0-13 Btm.
Flash Drum (O-13/O-12)
0-13 O.H.
Atm. Still (N-2)
73.5
NSB
IBP-350°F: 16.4
350-650°F: 51.4
650°F+: 5.8
N: 390 ppm
S: 140 ppm
API: 32.0
0-13 Btm. 104.4
Clean Oil Tank
58.4
VSOH
Vac. Still (N-3)
0.0
VSOH (Net)
M/U Oil 5.0
IBP-850°F: 3.6
850-975°F: 0.9
975°F+ 0.5
IBP-850°F: 57.7
850°F: 0.7
(Sample Only)
VSB
12.7
DAO
Rose-SR
IBP-975°F: 5.6
975°F: 11.8
IOM: 4.4
Ash: 11.6
Basis: 100 parts of Moisture-Ash Free Coal
IBP-850°F: 2.5
850-975°F: 5.9
975°F+ 3.6
IOM & Ash: 0.7
33.4
Rose Btm.
Drawing No.: PROSF012

FIGURE 5.8

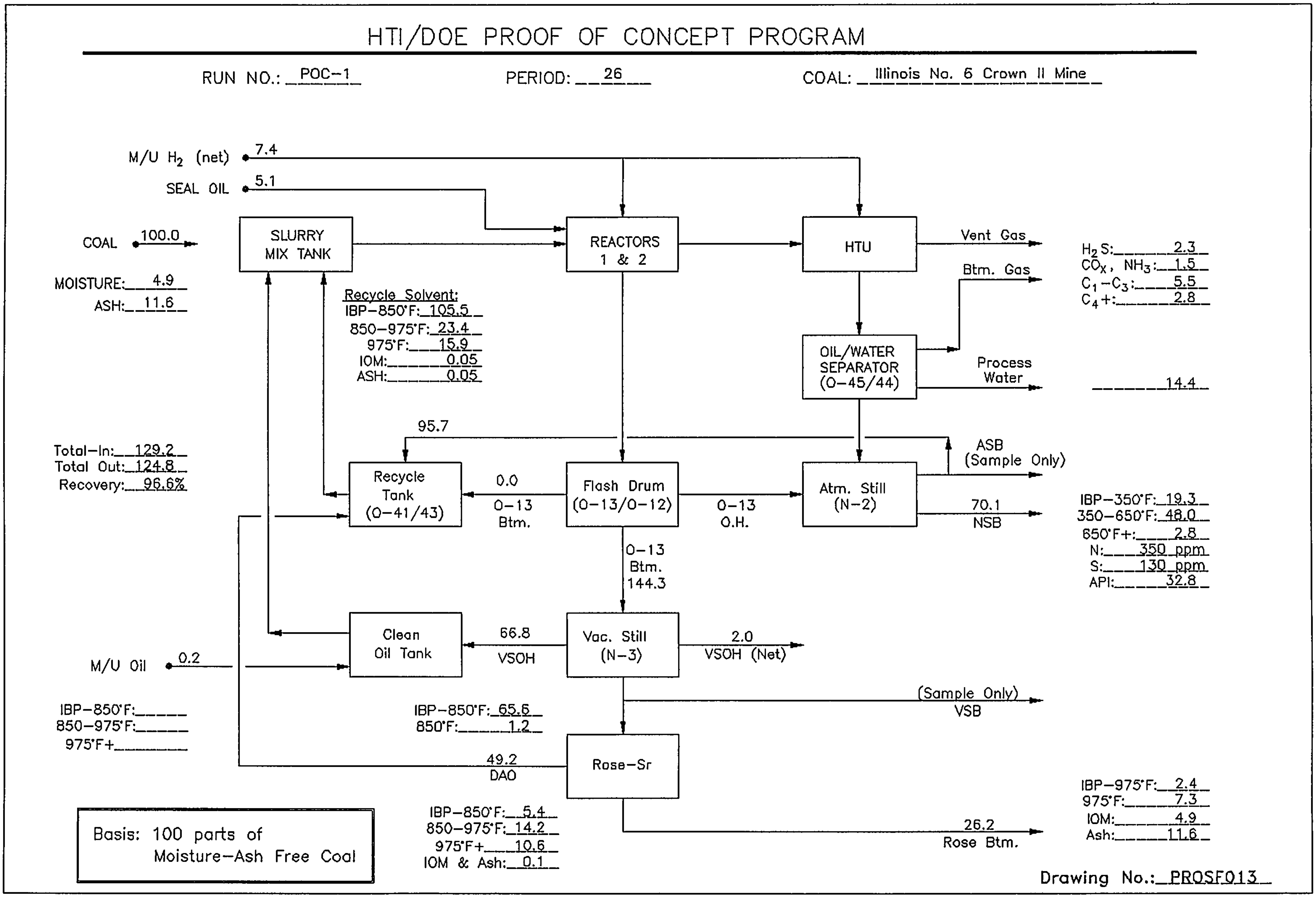

HTI/DOE PROOF OF CONCEPT PROGRAM
RUN NO.: POC-1
PERIOD: 26
COAL: Illinois No. 6 Crown II Mine
M/U H_2 (net) 7.4
SEAL OIL 5.1
COAL 100.0
MOISTURE: 4.9
ASH: 11.6
SLURRY MIX TANK
REACTORS 1 & 2
HTU
Vent Gas
Btm. Gas
H_2S: 2.3
CO_x, NH_3: 1.5
C_1-C_3: 5.5
C_4+: 2.8
Recycle Solvent:
IBP-850°F: 105.5
850-975°F: 23.4
975°F: 15.9
IOM: 0.05
ASH: 0.05
OIL/WATER SEPARATOR (O-45/44)
Process Water
14.4
95.7
ASB (Sample Only)
Total-In: 129.2
Total Out: 124.8
Recovery: 96.6%
Recycle Tank (O-41/43)
0.0
0-13 Btm.
Flash Drum (O-13/O-12)
0-13 O.H.
Atm. Still (N-2)
70.1
NSB
IBP-350°F: 19.3
350-650°F: 48.0
650°F+: 2.8
N: 350 ppm
S: 130 ppm
API: 32.8
0-13 Btm. 144.3
Clean Oil Tank
66.8
VSOH
Vac. Still (N-3)
2.0
VSOH (Net)
M/U Oil 0.2
IBP-850°F:
850-975°F:
975°F+
(Sample Only)
VSB
IBP-850°F: 65.6
850°F: 1.2
49.2
DAO
Rose-Sr
IBP-975°F: 2.4
975°F: 7.3
IOM: 4.9
Ash: 11.6
Basis: 100 parts of Moisture-Ash Free Coal
IBP-850°F: 5.4
850-975°F: 14.2
975°F+ 10.6
IOM & Ash: 0.1
26.2
Rose Btm.
Drawing No.: PROSF013

FIGURE 5.9

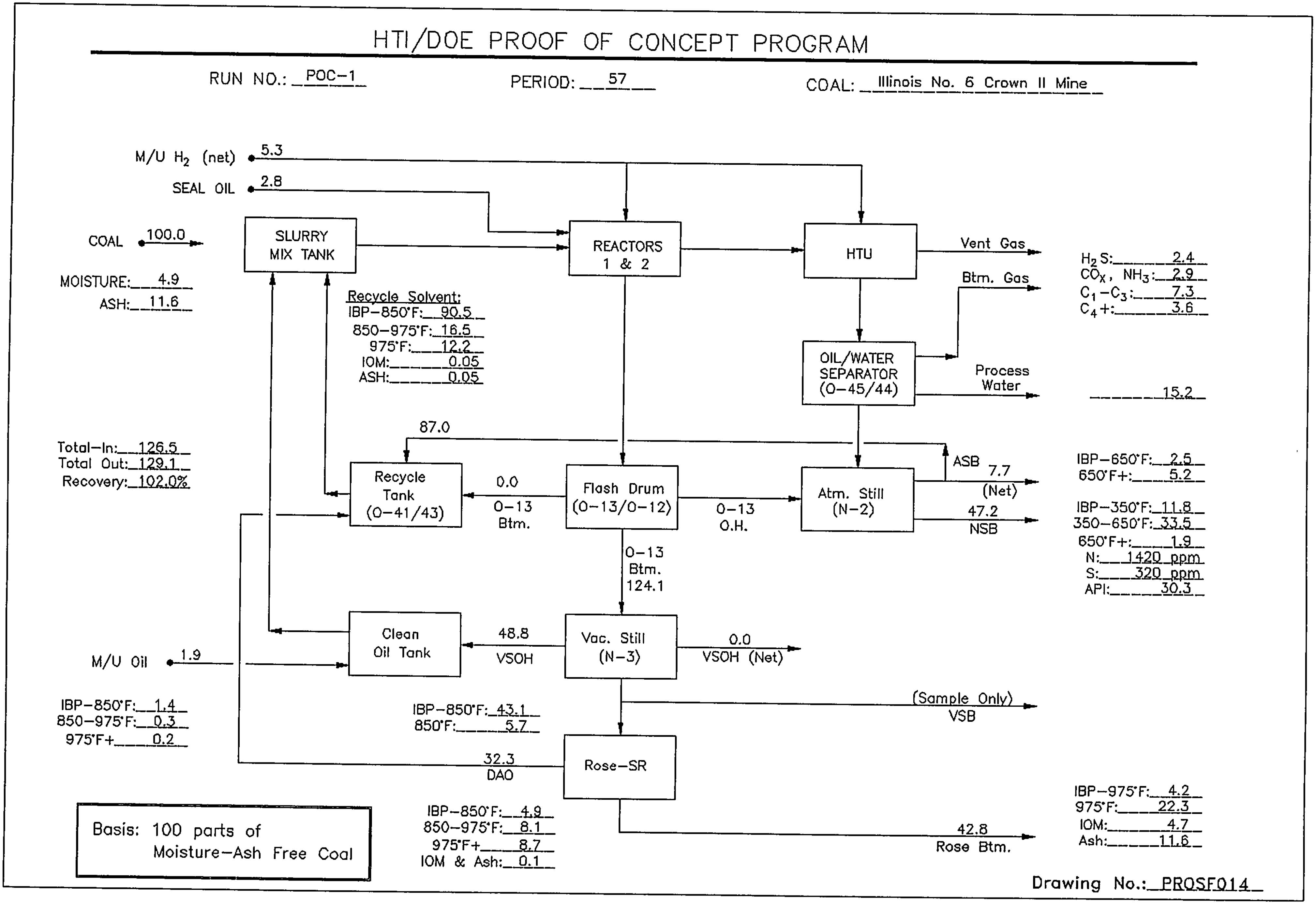

HTI/DOE PROOF OF CONCEPT PROGRAM
RUN NO.: POC-1
PERIOD: 57
COAL: Illinois No. 6 Crown II Mine
M/U H_2 (net) 5.3
SEAL OIL 2.8
COAL 100.0
MOISTURE: 4.9
ASH: 11.6
SLURRY MIX TANK
REACTORS 1 & 2
HTU
Vent Gas
Btm. Gas
H_2S: 2.4
CO_x, NH_3: 2.9
C_1-C_3: 7.3
C_4+: 3.6
Recycle Solvent:
IBP-850°F: 90.5
850-975°F: 16.5
975°F: 12.2
IOM: 0.05
ASH: 0.05
OIL/WATER SEPARATOR (O-45/44)
Process Water
15.2
Total-In: 126.5
Total Out: 129.1
Recovery: 102.0%
87.0
Recycle Tank (O-41/43)
0.0
0-13 Btm.
Flash Drum (O-13/O-12)
0-13 O.H.
Atm. Still (N-2)
ASB
7.7 (Net)
47.2 NSB
IBP-650°F: 2.5
650°F+: 5.2
IBP-350°F: 11.8
350-650°F: 33.5
650°F+: 1.9
N: 1420 ppm
S: 320 ppm
API: 30.3
0-13 Btm. 124.1
Clean Oil Tank
48.8 VSOH
Vac. Still (N-3)
0.0 VSOH (Net)
M/U Oil 1.9
IBP-850°F: 1.4
850-975°F: 0.3
975°F+ 0.2
(Sample Only) VSB
IBP-850°F: 43.1
850°F: 5.7
32.3 DAO
Rose-SR
IBP-975°F: 4.2
975°F: 22.3
IOM: 4.7
Ash: 11.6
Basis: 100 parts of Moisture-Ash Free Coal
IBP-850°F: 4.9
850-975°F: 8.1
975°F+ 8.7
IOM & Ash: 0.1
42.8 Rose Btm.
Drawing No.: PROSF014

FIGURE 5.10

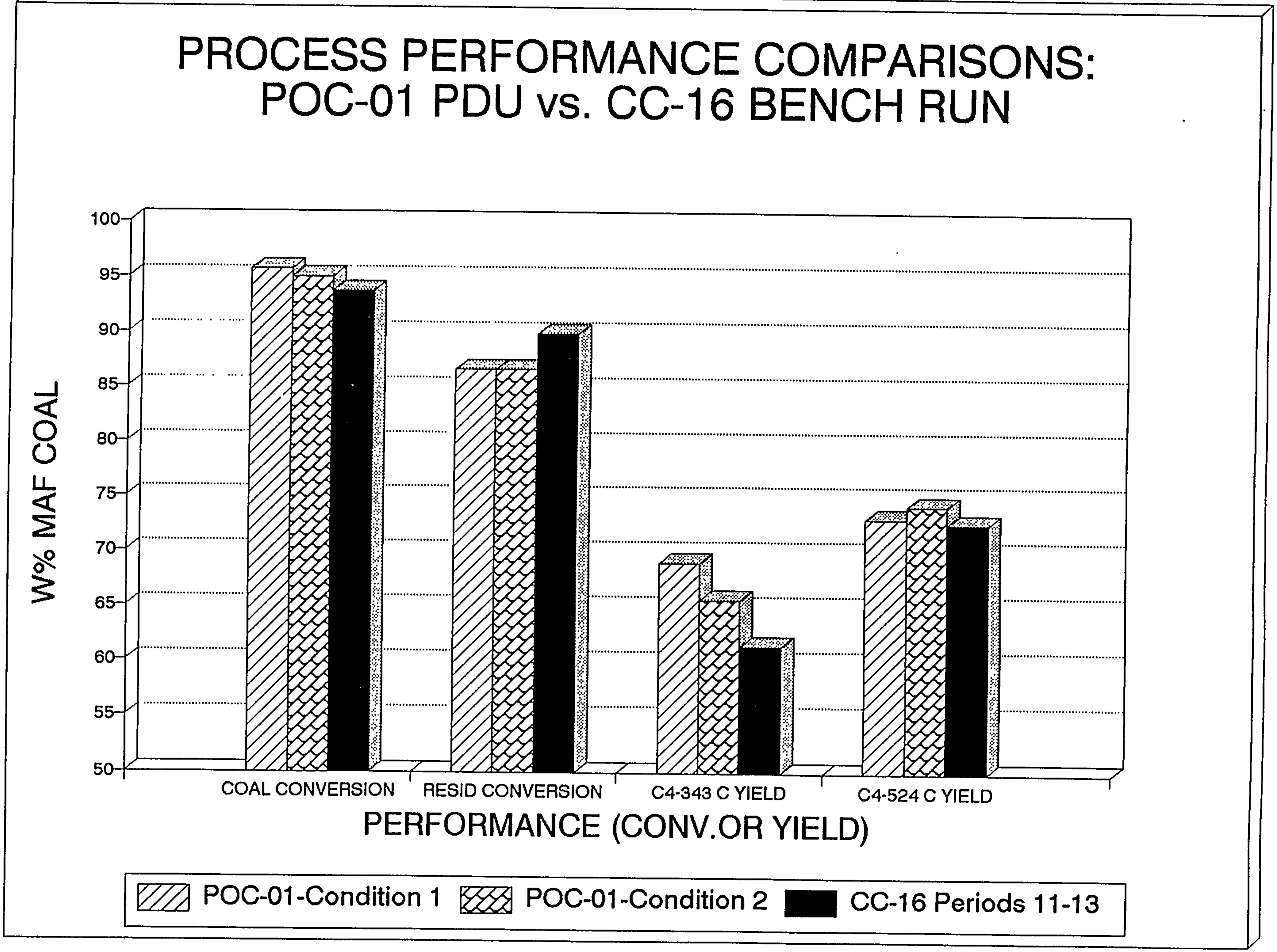

PROCESS PERFORMANCE COMPARISONS:
POC-01 PDU vs. CC-16 BENCH RUN
W% MAF COAL
100
95
90
85
80
75
70
65
60
55
50
COAL CONVERSION
RESID CONVERSION
C4-343 C YIELD
C4-524 C YIELD
PERFORMANCE (CONV.OR YIELD)
POC-01-Condition 1
POC-01-Condition 2
CC-16 Periods 11-13

FIGURE 5.11

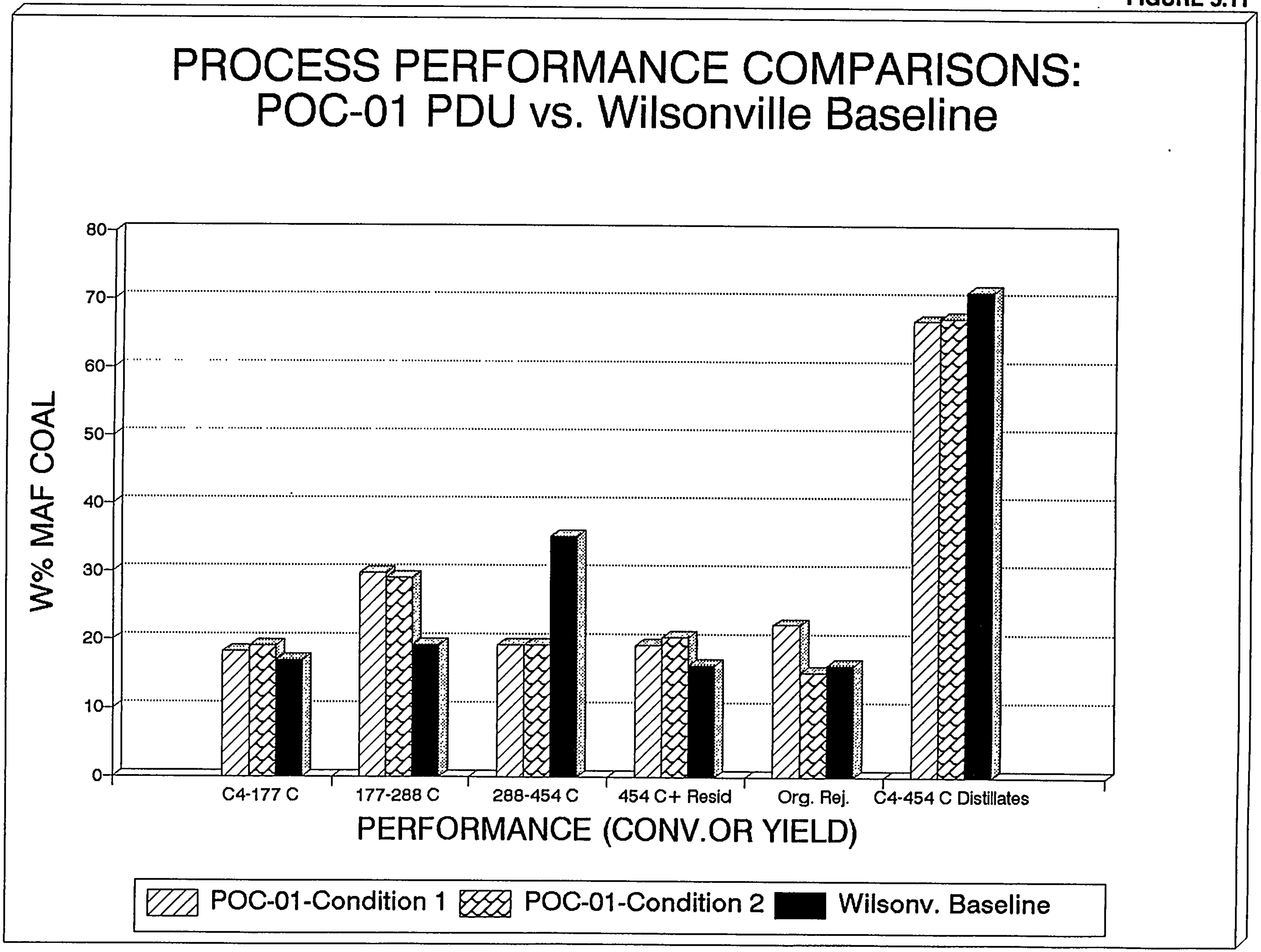
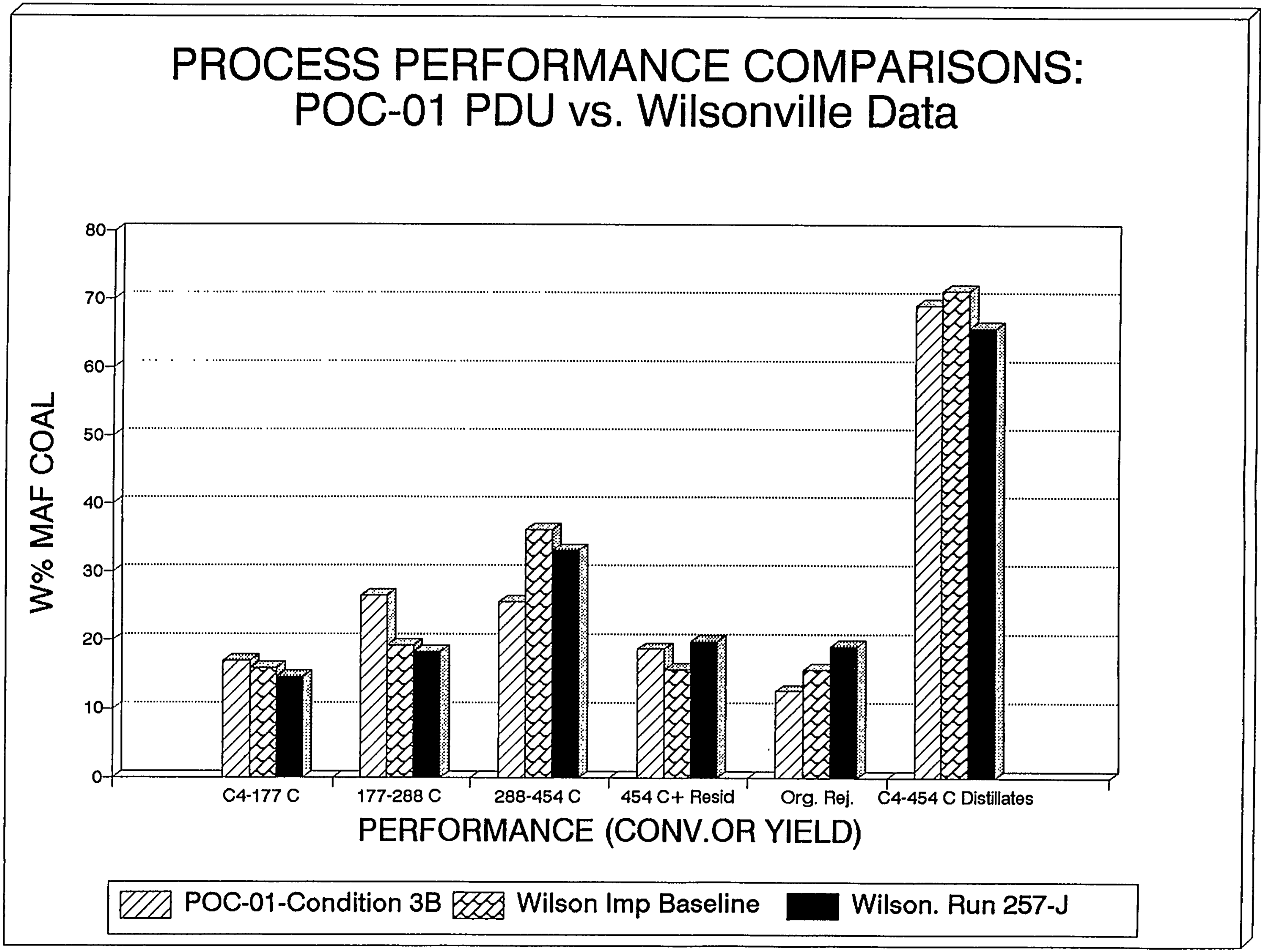

PROCESS PERFORMANCE COMPARISONS:
POC-01 PDU vs. Wilsonville Baseline
W% MAF COAL
80
70
60
50
40
30
20
10
0
C4-177 C
177-288 C
288-454 C
454 C+ Resid
Org. Rej.
C4-454 C Distillates
PERFORMANCE (CONV.OR YIELD)
POC-01-Condition 1
POC-01-Condition 2
Wilsonv. Baseline

FIGURE 5.12


PROCESS PERFORMANCE COMPARISONS:
POC-01 PDU vs. Wilsonville Data
W% MAF COAL
80
70
60
50
40
30
20
10
0
C4-177 C
177-288 C
288-454 C
454 C+ Resid
Org. Rej.
C4-454 C Distillates
PERFORMANCE (CONV.OR YIELD)
POC-01-Condition 3B
Wilson Imp Baseline
Wilson. Run 257-J

SECTION VI

DETAILED RUN ANALYSIS

A. CATALYST AGE AND INVENTORY

A.1. Catalyst Aging

The PDU operations are carried out under "equilibrated catalyst activity" conditions. To maintain a uniform activity of the catalyst in both the reactor stages, a periodic catalyst replacement schedule is implemented. Since both the reactor stages were charged with high activity fresh catalyst at the beginning, it was necessary to accelerate aging to approach equilibrium activity rapidly (first 15-20 days of operations on coal). Equilibrium catalyst activity is a function of catalyst replacement rate, as well as several other factors. Using the vast amount of data available from the bench scale operations, where catalyst undergoes batch deactivation (resid conversion activity vs. catalyst age), catalyst aging profiles for both reactor stages were predicted. For Illinois No. 6 coal operations, the derived catalyst batch deactivation factors were 0.012 fraction per day for the first stage catalyst and 0.060 fraction per day for the second stage catalyst (due to a higher temperature). During POC-01, different catalyst replacement rates were evaluated. *Table 6.1* gives the catalyst replacement schedule for the different POC-01 conditions. *Table 6.2* shows the estimated ratios of the equilibrium catalyst activity to fresh catalyst activity under different replacement rates and coal throughputs.

The projected resid conversion catalyst activity relative to fresh catalyst activity for both reactor stages is plotted on a daily basis in *Figures 6.1 (reactor K-1) and 6.2 (reactor K-2)*, using deactivation factors derived from bench scale batch operations and daily catalyst replacement rates (CRRs).

In CTSL operations, it is customary to define the catalyst age in terms of the amount of dry coal the catalyst has seen per unit weight of dry catalyst (fresh basis). The catalyst aging profiles in terms of g of coal/g of catalyst are shown in *Figures 6.3 and 6.4. Figure 6.3* shows this profile for different catalyst replacement rates and coal throughputs and also demonstrates the equilibration of the catalyst ages; Figure 6.4, an expanded version of Figure 6.3, demonstrates that after the first 20 days of the accelerated catalyst aging, the second stage catalyst deactivates (lower catalyst age) much more slowly than the first stage catalyst, mainly due to the higher catalyst replacement rate. This higher catalyst replacement rate also leads to a lower equilibrium catalyst age which is approached more quickly. The actual catalyst aging profile, in terms of g of coal/g of catalyst, during POC-01 PDU operations, is shown in *Figure 6.5*. The irregularities and humps in the aging profile are due to the shut-downs and startups that were encountered during the course of

POC-01. The actual aging profiles for both reactor stages match well with the predicted ones except for the final part (Period 49 onwards), due to the fact that catalyst replacement on a daily basis could not be effected during the final part of the run, as problems were encountered with the catalyst addition system. The performance of the catalyst in terms of process conversions and yields during POC-01, as it related to catalyst activity-aging behavior, is shown in *Figure 6.6*. As shown in this figure, there is a marked decline in the catalyst activity from Period 42-43 onwards, as exemplified by the decrease in resid conversion, distillate yield, and increase in the heavy resid yield.

A.2 Catalyst Daily Inventory and Mass Balance

Catalyst inventory calculations were made on a daily basis, using the catalyst addition and withdrawal schedules and the requisite analytical information on the catalyst withdrawals. This information included the toluene soluble oil-content of the spent catalyst charge, its weight loss upon ignition, and its metal (Mo and Ni) contents. The catalyst fines lost in the solid products of reaction (O-13 bottoms solids) were accounted for by the following catalyst daily mass-balance equation that was used to estimate catalyst inventory in the reactors on a fresh basis:

> Catalyst Inventory @ Day N = Catalyst Inventory @ Day (N-1) + Fresh Catalyst Added - Catalyst withdrawn (fresh basis) - Catalyst Fines lost in Solid Products (Fresh Basis)

The fresh catalyst contained 2.75 Wt% moisture. Attrition of the fresh catalyst to -20 mesh size fines was found to be, on average, below 3 W% of the catalyst makeup. Catalyst lost in the solid products was typically 0.2-0.4 lbs/day, accounting for less than 3 Wt% of the fresh catalyst added.

The daily catalyst inventory relative to the targeted catalyst inventory in both reactor stages is shown in *Figure 6.7*. As shown in this figure, reactor K-1 on average had about 95 Wt% of the targeted catalyst inventory, while reactor K-2 had close to the targeted inventory except for a few periods around Period 30.

A.3 Spent Catalyst Properties

Spent catalyst obtained from periodic withdrawals during POC-01 was typically washed thoroughly with toluene and then with acetone to wash off all the residual oil. The catalyst after washing, was dried and screened to separate +20 mesh size solids from the catalyst fines. Detailed analyses of the physicochemical properties of the catalysts, including surface areas, pore volumes, bulk densities, and elemental analyses are given in *Tables 6.3 and 6.4*. Daily catalyst withdrawals that were only washed with toluene and acetone were found to retain as much as

30 Wt% oil. Due to this, all of the work-up Periods' catalyst withdrawals were soxhlet extracted with toluene to remove all the soluble oil from the catalyst pores, and the detailed characterization was carried out on these catalysts.

As shown in *Table 6.4*, the carbon loading on the first stage withdrawn catalysts, soxhlet extracted with toluene, ranged from 11.3 to 13.8 Wt%. As expected, the second stage catalysts were noticeably higher ranging from a low value of 11.6% at the early stage of the run to a much higher value of 21.5% at the end of the run. Although the first stage catalysts contained less carbonaceous materials, metal contaminants (Fe, Ca, Na, Ti) loadings were higher than for second stage catalysts. The activities of withdrawn catalysts from several periods of this run, determined by microautoclave testing, are presented in Section VII Laboratory Support.

B. Solvent Composition

Under normal operation the slurrying oil was taken from the Recycle Weigh Drum (O-43). However, vacuum still overheads and makeup oil were also introduced through the Clean Oil Tank during the startup periods or when solvent imbalance occurred. As mentioned in the process description section, materials fed to O-43 included atmospheric still bottoms(ASB), deasphalted oil(DAO) from the ROSE-SR^{SM} operation or topped filtrate from the filter operation, and reactor liquid flash vessel bottoms under ashy recycle mode operation. Ashy recycle mode operation was implemented during all line-out periods and in Condition 1 in conjunction with the ROSE-SR^{SM} operation.

Composition of the slurrying oil is presented in *Figures 6.8 and 6.9*. With the exception of the line-out periods and Condition 1 (Periods 13-19), all other conditions were in the solids-free recycle mode of operation. The average composition is shown in the table below:

Condition	Periods	Days on Coal/Condition*	ASB	VSOH	DAO	Makeup Oil	O-13 Bottoms
1	14-19	4/2	20.3	17.4	11.4	11.9	39.0
2	20-26	11/2	40.8	37.9	18.2	3.1	0
3A	27-30	17/1	54.2	34.8	4.8	6.3	0
3B	42/43	4/2	47.1	28.3	9.2	15.4	0
4A/B	48/49	4/2	47.1	27.5	17.0	8.4	0
4C	55-57	5/2	44.9	31.1	16.6	7.4	0

* The first and second values represented the number of days on coal and into the specified condition, respectively.

In Condition 1, O-13 bottoms constituted the largest component of the recycle stream (39.0 Wt%) followed by ASB (20.3 Wt%) and VSOH (17.4 Wt%). The remaining material is split equally between DAO (11.4 Wt%) and makeup oil

(11.9 Wt%). For other conditions, about one quarter of the recycle oil was composed of DAO and makeup oil. The amount of makeup oil added to the process, which varied between 3.1 to 15.4 Wt%, was governed by the performance of the ROSE-SR^{SM} operation. As shown in *Figure 6.8*, the ROSE-SR^{SM} unit was not functioning properly through Condition 3A and most of the time in Period 3B.

The unreacted coal content in the line-out conditions and Condition 1 was less than 5.5 Wt%, while the maximum resid content ranged from 8.3 to 15.5 Wt%, as shown in *Table 6.5*.

In solids-free recycle operation, Condition 2 and 3 there was a maximum of 0.35 Wt% quinoline insolubles in the recycle solvent, reflecting the efficiency of the solids separation operation. The quality of solvent from selected periods, as determined by standard microautoclave test procedure under a non-hydrogen environment, is shown in *Table 6.6*. There seems to be no significant correlation between the THF coal conversion and the properties of the solvent.

C. PRODUCT QUALITY

The product streams from the PDU Operations are vent gas, bottoms gas, sour water, naphtha stabilizer bottoms and ash reject (ROSE-SR^{SM} bottoms). The analysis of these streams and several internal streams (atmospheric still bottoms, vacuum still overheads, feed slurry, recycle oils and reactor liquid flash drum bottoms) are attached in Appendix D.

The qualities of selected product and internal streams are discussed in this section.

C.1 Naphtha Stabilizer Bottoms

Naphtha stabilizer bottoms (NSB), the overhead stream from the Atmospheric Still Column, was slated to be the sole liquid product stream under resid extinction mode of operation in POC-01. It should be noted however at this point that, in an ideal extinction recycle mode of operations, all the material, boiling above 343°C, gets recycled into the process. This means, most of the vacuum still overheads and atmospheric still bottoms are recycled with the other heavier internal streams such as O-13 bottoms and the deasphalted oil. As seen from Figures 5.6 through 5.9, this was not really the case during POC-01 and indeed, net liquid products in the boiling range of 343-524°C were also formed due to contributions from the unrecycled parts of the ASB and the VSOH streams. This effect can also be seen in Tables 5.1, 8.2, and 8.3. During Condition 3, in which the coal feed rate was increased from 70 kg/h to 89.0 kg/h, there was 1.5 to 3.0 Wt% water present in the NSB. The problem was resolved for future PDU runs by increasing the size of the water/oil separator to provide more residence time for the disengagement of water.

In POC-01 the product recovery section of the PDU was configured so that the feed to the Atmospheric Still was composed of products from the Hydrotreater (which was internally bypassing material and so not performing as expected) and the Overheads (O-12) from the Reactor Liquid Flash Drum (O-13). The latter product, approximately 20-25 Wt% of the feed to the column, was not hydrotreated.

NSB quality was not strongly influenced by changes in process severity. The most influential factor was the catalyst replacement rate. NSB quality took a drastic downturn in Condition 4, during which there was no catalyst replacement in either reactor. The NSB target cut point was 343°C (650°F). However, in most cases the end point was in the range of 350-380°C (660-716°F), reflecting the efficiency of the fractionation operation, as shown in *Tables D.1a to D.1c.* The main product was separated into four boiling point fractions according to the ASTM D-86 Distillation procedure. The weight distribution of these fractions was in the range of:

Light Naphtha	(IBP-177°C)	22.2-25.7 Wt%
Heavy Naphtha	(177-288°C)	37.1-46.5 Wt%
Light Distillate	(288-343°C)	23.1-35.4 Wt%
Heavy Distillate	(343°C+)	2.7-8.3 Wt%

as shown in *Table 6.7*

NSBs from Conditions 1 to 2 contained over 62 Wt% of these materials boiling below 288°C (550°F). These materials were also rich in hydrogen, ranging from 12.4 to 12.7 Wt%, and contained less than 140 and 420 ppm of sulfur and nitrogen, respectively. (Sulfur determination were performed on caustic washed samples.) Starting with Period 43 (Condition 3B), hydrogen content declined gradually to 11.8-11.9 Wt% towards the end of the run, while nitrogen and sulfur increased by 200 to 400 Wt%, as illustrated in *Figures 6.10 and 6.11 and Table 6.8*. The distributions of the nitrogen and sulfur in each boiling point fractions are shown in *Figure 6.11*.

C.2 Sour Water

In addition to water associated with moisture in the coal and generated from hydrogenation of oxygen in coal, water was injected downstream of the hydrotreater to avoid salt build-up. As part of the elemental balance requirement, nitrogen and sulfur in the sour water stream were determined on a regular basis.

The sour water stream contained dissolved ammonia and hydrogen sulfide. Typical nitrogen and sulfur contents were 2-4 Wt% and 1-2.5 Wt%, respectively, as given in *Table 6.9.*

A special sample of sour water was collected in Period 44 and analyzed by Environmental Science & Engineering, Inc. of Plymouth, Pa. The results of these analyses are shown in *Table 6.10* along with the allowable limit for each category. This water sample was slightly basic with a pH value of 8.67. The concentrations of total organic carbon and phenols were significant at 2,530 and 1,290 mg/l, respectively. Also, the BOD requirement was high at 15,200 mg/l. The phenol content, BOD and COD levels significantly exceed their respective limits. Therefore, sour water must be treated to meet these limits.

The types of phenolic compounds in the sour water were determined using a GC-MS technique by the Core Laboratories, Corpus Christi, Texas. A summary report by Core Laboratory is attached in *Appendix G.*

C.3 Reactor Liquid Flash Drum Bottoms (0-46)

Slurry product from the 0-1 Hot Separator was flashed in the Reactor Liquid Flash Drum (0-13). The bottoms from 0-13 became the feed to the recycle liquid/solids separation system. In POC-01 the 0-13 bottoms stream (also called 0-46 slurry) served as a tie between the coal liquefaction and the solids separation sections. For this reason 0-46 slurry was characterized in detail. Based on the analysis of 0-46 slurry, the performance of the coal liquefaction and the solids separation systems were determined.

The composition of 0-46 slurry is summarized in *Table 6.11*.

With the exception of Period 43, which had higher than expected coal and ash contents, there was 3.94 to 4.64 Wt% unreacted coal in 0-46 slurry. The highest level of ash (11.2-12.1 Wt%) was observed in Condition 1, when ashy recycle mode was used. The ash level in 0-46 slurry dropped approximately 25% to 9.57 and 9.08 Wt% in Periods 24 and 26 (Condition 2), when solid-free recycle mode was implemented. This value rose gradually to 11.6 Wt% as the recycle solvent/coal ratio was reduced from 1.2 to 0.9 towards the end of the run.

The quality of 0-46 slurry followed a trend similar to that for the naphtha stabilizer bottoms. The H/C ratios were in the same range as the deasphalted oil throughout the run. The H/C ratio was high, around 1.34 to 1.36, in Conditions 1 and 2 and declined thereafter to a low level of 1.08-1.11 at the end of the run. The decline in quality was generally due to an increase in coal processing rate and no catalyst replacement from Period 45 onward. The 524°C (975°F) resid content followed a similar trend. 0-46 slurry contained 14.0 to 15.5 Wt% solids-free resid at the beginning of the run (Conditions 1 and 2) and increased to a higher range of 22.9-28.7 Wt% in the second half of the run (Conditions 3 and 4), as illustrated in *Figure 6.12*.

Detailed analyses of 0-46 slurry are given in *Tables D.2 and D.3*.

C.4 Atmospheric Still Bottoms and Vacuum Still Overheads

Atmospheric still bottoms (ASB) was a major component of the recycle solvent, especially during solids-free recycle mode of operations, as discussed earlier in this section (Solvent Composition). The relative proportion of ASB in the recycle solvent is compared with the concentration of vacuum still overheads (VSOH) in the table below:

Wt% ASB and VSOH in Recycle Solvent		
Periods	**ASB**	**VSOH**
14-19	13.2-26.9	10.3-30.4
21-26	37.5-46.4	34.7-40.3
27-31	39.6-68.8	31.1-38.6
41-43	42.6-51.6	15.4-28.5
58-50	44.7-51.5	19.4-27.2
54-58	30.5-56.2	20.6-32.8

The API gravity of ASB ranged from 17.3 to 21.3° through most parts of the run (Period 1 to Period 50) and declined to 13.5-14.4° during the last operating condition (Periods 51-58). The performance of the Atmospheric Still was very constant throughout the run. The amount of 343°C- (650°F-) distillate varied within a narrow range of ±7.0 V% of an average value of 28.5 Vol%. The initial boiling point and end point were in the range of 199-281°C (390-538°F) and 450-480°C (842-896°F), respectively. Detailed boiling point distributions (determined by the ASTM D1160 distillation procedure), API gravities and elemental analyses of selected periods are given in *Tables D.4a & D.4b.* This stream was very rich in hydrogen and low in heteroatoms. In Periods 9 to 43, the hydrogen content ranged from 11.7 to 11.3 Wt%, while it dropped to a lower range of 10.2 to 11.0 Wt% in the later periods.

The vacuum still overheads were recycled through the Clean Oil Tank. With the exception of Period 19, which had an initial boiling point (IBP) of 253°C (487°F), the IBP for other periods varied slightly between 271-279°C (520-534°F) and contained less than 17.0 Wt% boiling below 343°C (650°F). *Tables D.5* presents the boiling point distributions and elemental analyses of VSOH collected from the workup periods of each process condition. As anticipated, the hydrogen content of VSOH was generally lower than that of ASB, ranging from 11.1 to 11.3 Wt% in Period 4 to 43 and declining in quality in Periods 49 to 57.

C.5 Inspection of First Stage Slurry Samples

Samples of the first stage slurry were obtained during Period 40A and 42B of POC-01 (Run 260-04). The analyses of the two second stage products (O-46 samples from Periods 26 and 49), that were before and after these Periods, were also provided in *Table 6.12*. The analyses were conventional D-1160 distillation characterization and elemental analysis of the filterable portion. The filter solids were characterized as ASTM ash, and unconverted coal (the balance of the quinoline insoluble portion). Elemental analyses were also obtained on the filter solids.

First stage coal conversion for Periods 40 and 42 was 88.7 and 90.8 Wt% maf coal, respectively. These values were about 92 to 95% of the overall coal conversion after the second stage. The H/C ratio of the first stage samples, both the filtrate and the filter solids, were in general higher than these of the second stage samples. In the low-high temperature sequence the first stage is more favorable for hydrogenation reactions, facilitating the transfer of gaseous hydrogen to the solvent and coal.

C.6 Analysis of TBP Product Fractions

The net process distillates (naphtha stabilizer bottoms) from two representative steady-state periods of each of the operating conditions from POC-01 were blended together for detailed characterization. Four blended samples were prepared as follows:

Sample No. 1	Periods 15 and 17
Sample No. 2	Periods 24 and 26
Sample No. 3	Periods 48 and 50
Sample No. 4	Periods 56 and 57

Using a packed fractionation column, these samples were cut into four true boiling point (TBP) fractions (the same fractions as the yield structure), IBP-177°C (IBP-350°F), 177-288°C (350-550°F), 288-343°C (550-650°F), and 343°C+(650°F).

As shown in *Tables 6.13a to 6.13d and 6.14a to 6.14d*, the properties of the TBP fractions were similar for the first two operating conditions (samples 1 and 2), but differed for the last two operating conditions (samples 3 and 4). This difference in properties is very prominent for the individual heteroatom contents of the TBP fractions. Samples 3 and 4 were obtained from periods of POC-01 when fresh catalyst addition to both the reactors was not operative. As a result of low catalytic activity in the reactors, the overall quality of the net process distillates deteriorated during these operating periods. For the first two samples, the gasoline and diesel cuts of the TBP fractions have good quality. e.g., an octane number of over 60 for the gasoline cut and a cetane number of close to 35 for the diesel fraction. Sulfur and nitrogen contents of these TBP cuts are also within the specs for gasoline and diesel. A mild hydrotreatment for the diesel cut and reforming for the gasoline cut would be needed at most to boost the quality of these TBP fractions to meet the specifications for the premium transportation fuels. High hydrogen contents (12-14 Wt%) have also been obtained for both gasoline and diesel cuts of the TBP fractions, with low heteroatom contents (especially for the first two samples), indicating significant hydrogenation/hydrocracking even in the absence of an in-line hydrotreater.

D. ROSE-SR^{SM} Solids Separation Unit

Prior to the start of POC-1, HRI installed a ROSE-SR^{SM} solids-separation unit licensed by Kerr-McGee Corporation as part of the Proof-of-Concept direct coal liquefaction facility. Major equipment for this unit was obtained from the Wilsonville Advanced Liquefaction facility; however, extensive new equipment was added and the flow scheme was modified. The POC ROSE-SR^{SM} unit as shown in Figure 4.3 was designed to use a pentane solvent in place of toluene and mixed solvents employed at Wilsonville. The Lighter pentane solvent is preferred due to the improving quality (lower preasphaltene) of the resid with the latest Catalyst Two-Stage Liquefaction (CTSL) Technology.

Also, the third settler stage used at Wilsonville was eliminated, providing a single liquid deasphalted oil (DAO) product. The ROSE-SR^{SM} unit feed is the vacuum tower bottoms which is nominally an 454°C+ (850°F+) slurry stream. The purpose of the ROSE-SR^{SM} unit is to separate solids (ash and unconverted coal) from liquefaction bottoms to recover a solids free recycle oil for coal liquefaction and to reject a solids-containing product. When operating properly, a fine powder solid product can be produced. This ash concentrate product can be used as feed for gasification (for hydrogen or fuel gas production) or for combustion (for steam or power generation).

The objectives for the ROSE-SR^{SM} unit for POC-1 were:

- To commission the newly installed equipment and demonstrate operability with a pentane solvent.

- To demonstrate continuous operability on a coal liquefaction bottoms slurry (Wilsonville operated the ROSE-SR^{SM} unit batch-wise).

- To demonstrate operability in a CTSL Process resid extinction-recycle mode of operation.

- To obtain maximum recovery of resid with minimum energy rejection to the ash concentrate. (Target ≤ 15% energy rejection with Illinois coal CTSL Operations)

- To achieve a solids-free (≤ 1W%) deasphalted oil for recycle.

D.1 Performance of the Off-line Tests (Nov 20 to Dec 1 1993)

The ROSE-SR^{SM} unit was initially brought on-stream on November 20 (Period 10 shutdown) for off-line tests to confirm equipment operability and to determine preferred operating conditions to produce a flowable solids product.

During the Period 10 unit shut down several off-line tests were performed. Results of two material balance periods are discussed in this report. The duration of these tests were:

Test I 11/20/93 1900 hour to 11/21/93 0100 hour
Test II 11/30/93 2200 hour to 12/01/93 0430 hour

A mass balance for each test period was determined based on the weight of the solid rejects (ROSE-SR^{SM} bottoms) and the cumulative level changes in Tank O-61 (ROSE-SR^{SM} feed) and O-65 (deasphalted oil). Samples of all three streams were taken and analyzed. The ROSE-SR^{SM} feed sample was collected from the pump discharge of the recirculation loop, while the Deasphalted oil (DAO) sample was taken downstream of Tank O-65 and upstream of LCV-933. The solid reject sample was collected from the top of the drum at the end of the test periods.

Relatively stable operations were established at a feed rate of 42.2 to 44.5 kg/h. The material balance was 104 and 107% for Test I and Test II, respectively (*see Table 6.15*). Powder ash rejects, which contained less than 33.3 Wt% quinoline soluble materials, were collected. However, the amount of recovered solids, quinoline insolubles in DAO and ash rejects were higher than in the feed, suggesting part of the solids were from operations prior to these material balance periods. The other possibility was that the ash reject samples collected from the top of the drum were not representative for the whole test periods.

The unreacted coal to ash ratios of the feed to the ROSE-SR^{SM} unit were essentially the same as those of the bottoms, as shown in the table below, suggesting that there was no degradation of these materials in the ROSE-SR^{SM} unit.

UNREACTED COAL/ASH RATIO

	TEST I	TEST II
Feed	0.42	0.71
Bottoms	0.41	0.68

In these off-line tests operating temperature and solvent/feed ratio were established. The unit was brought on-line from Period 14 onward.

D.2 Performance of the Integrated Operations

Starting from Period 14 the Residuum Oil Supercritical Extraction - Solids Rejection (ROSE-SR^{SM}) unit was brought on-line as an integral part of the liquefaction operations. Deasphalted oil (DAO) from the ROSE-SR^{SM} was recycled, while the unreacted coal and mineral matter were rejected as a solids-rich bottom stream.

N-pentane was used as the solvent throughout the entire run. Material balances for selected periods are shown in *Table 6.16.* and *Figure 6.13.* With the exception of Period 43 and 47, the overall recovery was within 95-105 Wt%. However, there were more variances in the ash recovery. These variances were mainly due to the sampling technique used in collecting the bottoms. Most of the bottom samples were taken from the top of the collection drum. The degree of deviation from 100% recovery is a reflection on the consistency of the quality of the bottoms across the drum.

Results of the TGA and compound class type (determined by solvent extraction) of the Feed, Bottoms and DAO are given in *Tables 6.17 and 6.18.* The majority of the feed to the ROSE-SR^{SM} unit contained 19 to 30 Wt% of boiling below 524°C (975°F), while the solid content (quinoline insolubles) ranged from 24.0 to 37.3 Wt%. There were significant changes in the content of the asphaltenic/preasphaltenic materials (pentane insoluble, quinoline solubles) in the feed as the run progressed. In the first half of the run, Periods 14 to 29, the asphaltenic/preasphaltenic content was mostly below 20 Wt% but increased to 30-40 Wt% during the last several days of the run, when catalyst addition was not conducted.

Unit performance in term of rejecting solids improved through the course of the run. During the initial phase of integrated operations, a considerable portion of the solids remained in the DAO. The solid concentration in the DAO varied from 3.3 to 20.7 Wt% in Periods 14 to 21. During the latter part of the operations, from Period 22 onward, the majority of the solids ended up in the bottom stream. The solid content of the DAO was mostly below 2 Wt%, with the exception of Periods 48 and 49 which showed 2.4 and 3.0 Wt%, respectively.

The amount of pentane solubles lost through the bottoms also decreased with operating experience. Towards the end of the run, the ash rejects was composed of mostly n-pentane insoluble (PI) materials. The soluble fraction was less than 9 Wt% and was as low as of 1.8 Wt% in Period 50. The production rate of bottoms in relationship to the level of pentane insolubles in the feed to the ROSE-SR^{SM} unit is plotted in *Figure 6.14.* Also, based on Kerr-McGee's experience in solid rejection, it was anticipated that the amount of resid required to agglomerate the solid material would be roughly 1/3 the weight of the solids. In this regard n-pentane was the proper solvent for the first half of the run in which the content of the asphaltenic matter was below 20 Wt% in the feed to the ROSE-SR^{SM} Unit. However, as the amount of the asphaltenic material increased, as happened in the second half of run, the amount of asphaltenic material rejected through the bottoms was much higher than the amount required for agglomerating the solid residuals. As a consequence the liquefaction performance of the latter periods was severely reduced. In order to raise the distillate yields, a strong ROSE-SR^{SM} solvent will be required to recover these excess asphaltenic materials for reprocessing to extinction in the process.

A series of parity plots are given in *Figures 6.15-6.17* illustrating the effectiveness of rejecting pentane, toluene and quinoline insolubles through the ROSE-SR^{SM} bottoms compared to feed values.

D.2a Organic and Energy Rejections

The organic and estimated energy rejections are listed in *Table 6.19.* With the limited operating experience with the ROSE-SR^{SM} system, the lowest organic (12.6 Wt% maf coal) and energy (12.5 Wt% coal) rejections were attained in Period 43. Although in Periods 56 and 57 the unit operation was very smooth, high rejections (30-34 Wt% maf coal) were experienced. This is because of the high rejection of asphaltenic materials, as discussed above. Also shown in *Table 6.19* are the coal conversions for the same periods. These conversion levels were very comparable with values obtained by analyzing the vacuum tower feed and reflect the integrity of the solid separation system. There was no noticeable degrading of the product in the solids separation system as was experienced previously at the Wilsonville Advanced Liquefaction Facility using a heavier solvent.

D.2b Analysis of Core Sample

A core ash concentrate (O-63) sample was taken from Periods 19 and 26 to determine the average properties of the ash reject. The sample from Period 19 contained 47.6 Wt% unreacted coal and ash, while the solid content of the core sample from Period 26 was 15.3 Wt% higher as shown in *Table 6.20*. As a result, the measured heating value was 24.68 x 10^6 and 19.89 x 10^6 Btu/lb for the former and latter samples, respectively. These values are approximately 14% lower than for the feed coal. However, the ash rejects contained less sulfur, 2.8-3.3 Wt% as compared with 3.9 Wt% in the original coal.

The appearance of the ash reject varied with oil and resid content. The reject obtained from Period 26 was a free-flow powder consisting of 42 Wt% particles less than 40 mesh. The 40 mesh plus material was a loosely packed agglomerate that disintegrated easily upon impact.

D.2c Summary of ROSE-SR^{SM} Operations and Performance

The ROSE-SR^{SM} unit was successfully commissioned and operated during POC-01. Both the off-line tests and on-line service resulted in effective rejection of unreacted coal and mineral matter generated from the two-stage liquefaction of Illinois coal. Within a relatively short operating duration, the performance obtained from the ROSE-SR^{SM} unit at HRI, in term of solids and energy rejection efficiencies, was comparable with results achieved by the Wilsonville operation.

From an operations point of view, a combination of inexperience and direct coupling with an experimental pilot unit posed a great challenge. Each change of condition in the coal liquefaction section required a great effort for the operating staff to fine tune the unit to a new set of operating variables for effective removal of solids in a powdery form. In a long duration demonstration run with the proper solvent, the ROSE-SR^{SM} can serve as an effective mean of rejecting solids from coal liquefaction.

E. On-line Hydrotreater

In Periods 1 to 27, a trickle bed hydrotreater was connected to the overheads stream of the Hot Separator. The overheads stream consisted of low boiling distillate, gases, steam and excess hydrogen from the second stage reactor, This stream was further upgraded in the hydrotreater packed with Criterion 411 Ni/Mo extrudate catalyst.

The on-line hydrotreater was in service for part of the run and was taken out of service after it was determined that possible internal bypassing had occurred.

The nitrogen and sulfur contents of the hydrotreater distillate (O-5) are given below:

Period	Nitrogen [ppm]	Sulfur [ppm]	Hydrogen [Wt%]
Hydrotreater in Service			
9	246	89	12.55
13A	394	157	
14A	360	149	12.44
14B	374	139	12.45
15B	505	130	
19B	449	150	
Hydrotreater off service			
16B	485	137	12.27
17A	636	111	12.02

The quality of the separator overheads did not improve significantly in the presence of the hydrotreater, suggesting possible internal bypassing. The nitrogen content varied from 246 to 505 ppm with the hydrotreater in-line and was only 1.2-2.5 times higher than the values observed in Period 17, as illustrated in *Figure 6.18*. Surprisingly, the sulfur content was even lower with the hydrotreater out of service. The heteroatoms level was more than 15 times higher than values obtained from the laboratory scale tests under similar operating conditions using the same catalyst. Details of the laboratory scale tests are discussed in Section VIIC.

Table 6.1
Catalyst Replacement Schedule

Periods	Coal Space Velocity Lbs/hr/ft^3	K-1 CRR Lb/T	K-2 CRR Lb/T
1-19	20	0.25	0.5
		[Alternate days replacement]	
20-42	20-30	0.75	1.5
43-46	20-30	0.5	1.0
47-58	30	0	0
		[Catalyst addition problems]	

Table 6.2.
Predicted Equilibrium Catalyst Activity

Catalyst	Coal Space Velocity Lbs/hr/ft^3	CRR Lb/T	Equilibrium Fresh Catalyst Activity Ratio
K-1	20	0.5	0.56
K-1	30	0.75	0.74
K-2	20	1.0	0.34
K-2	30	1.5	0.54

TABLE 6.3

POC-01 Analyses of Withdrawn Catalyst (Bulk Washed Procedure)

First Stage Catalyst

Period	Fresh	10	16	20	24	26	37	43	Shutdown
Bulk Density, g/cc	0.872	0.788		0.945		0.878			0.859
Particle Density, g/cc		1.216	1.180	1.432		1.332		1.436	
Ignition Loss, W%	3.08	22.57	19.71	32.66	26.58	26.35			17.08
Elemental Analysis, W%									
Carbon		18.79	14.56	24.89	23.96	20.51	15.67		
Hydrogen		1.04	0.96	2.22	2.00	1.69	1.36		
Nitrogen		0.28	0.17	0.08	0.14	0.09	0.16		
Sulfur		6.26	6.48	5.14	4.27	4.79	5.48		
Major Metals									
Molybdenum	12.25	4.71	7.32	6.84	6.39	6.72	7.02	6.13	6.52
Nickel	2.60	1.04	1.74	1.38	1.36	1.56	1.60	1.42	1.38
Iron	0.01	0.14	0.16	0.24	0.32	0.34	0.49	0.45	0.66
Sodium	0.07	0.32	0.72	0.92	0.82	0.82	0.75	0.56	0.64
Calcium	0.00	0.06	0.05	0.05	0.28	0.06	0.05	0.06	0.10
Total Contaminants, W%		24.09	20.47	31.77	30.52	26.69	22.04		
Toluene Soluble Oil, W%				17.66	14.41	27.80	5.24	12.02	36.30

Second Stage Catalyst

Period	9	15	19	24	26	37	43	Shutdown
Bulk Density, g/cc	0.957		1.047		0.807			0.923
Particle Density, g/cc	1.438	1.325	1.518		1.187		1.486	
Ignition Loss, W%	32.73	29.94	39.65	32.33	23.14			
Elemental Analysis, W%								
Carbon	28.45	24.25	32.47	28.04	16.75	24.29		
Hydrogen	2.70	1.85	2.94	2.12	0.98	1.15		
Nitrogen	0.12	0.13	0.08	0.13	0.19	0.17		
Sulfur	5.26	5.36	4.64	4.52	5.50	4.77		
Major Metals								
Molybdenum	6.18	5.36	6.38	6.65	8.14	7.34	5.99	7.73
Nickel	1.11	1.20	1.38	1.38	1.66	1.59	1.40	1.56
Iron	0.04	0.41	0.04	0.05	0.07	0.07	0.08	0.16
Sodium	0.73	1.09	0.95	1.04	1.08	0.79	0.58	1.04
Calcium	0.04	0.05	0.03	0.03	0.03	0.22	0.05	0.05
Total Contaminants, W%	35.26	31.47	22.65	34.43	22.65	29.75		
Toluene Soluble Oil, W%	21.43		24.66	16.38	29.26	11.82	19.98	26.80

TABLE 6.4
POC-01 Analyses of Withdrawn Catalyst
(Toluene Extracted Using Soxhlet Apparatus)

First Stage Catalyst

Period	10	20A	24	37	43	S/D
Elemental Analysis W%						
Carbon		11.51	12.55	11.19	13.76	11.30
Hydrogen		0.59	0.63	0.55	0.59	0.60
Nitrogen		0.20	0.17	0.19	0.23	0.14
Sulfur		5.98	5.68	5.85	5.50	5.81
Eq. Fresh Cat., W%						
Basis: Contaminants		82.3	81.1	82.9	80.7	72.9
Basis: Mo Content		67.8	60.9	60.5	56.9	53.2
Basis: Ni Content		64.5	61.1	64.9	62.1	53.0

Second Stage Catalyst

Period	9	19	24	37	43	S/D
Elemental Analysis W%						
Carbon	11.60	14.28	15.97	15.11	15.50	21.50
Hydrogen	0.40	0.64	0.64	0.64	0.58	0.65
Nitrogen	0.18	0.17	0.15	0.20	0.22	0.15
Sulfur	6.43	6.20	5.50	5.59	5.48	5.66
Eq. Fresh Cat., W%						
Basis: Contaminants	82.9	79.5	81.1	79.3	79.5	61.5
Basis: Mo Content	64.2	69.1	60.9	68.0	61.1	63.1
Basis: Ni Content	54.3	70.4	61.1	69.4	67.3	60.0

TABLE 6.5

Estimated Composition of Recycle Solvent

Condition	Period	IBP-524°C	524°C+ Resid	Unreacted Coal	Ash
L/O	1	86.17	8.32	2.75	2.76
	2	--	--	3.40	4.50
	3	77.31	10.87	5.15	6.67
	4	79.24	9.11	4.33	7.32
	6	87.50	6.30	2.98	3.14
	7	--	--	--	6.78
	8	69.18	13.23	5.27	12.3
	9	74.49	8.83	4.62	3
					12.0
	12	73.46	14.71	4.94	6
	40	78.52	10.04	3.66	6.89
	46	73.45	14.29	3.62	7.78
					8.64
1	17	69.15	15.54	4.10	11.2
	19	80.75	10.48	2.43	1
					6.34
2	26	88.96	10.93	0.03	0.08
3B	43	93.6	6.09	0.08	0.23
4C	57	79.5	10.07	3.81	6.62

TABLE 6.6

POC-01 Solvent Quality Tests

Period No.	Condition No.	Days At Condition	THF Conversion [Wt% Maf Coal]
Start-up Solvent	L-803 L-809	1st Batch 2nd Batch	69.4 77.0
4	L/O	4	60.2
9	L/O	4	61.5
12	L/O	2	61.7
17	1	5	64.0
19	1	7	62.0
24	2	5	63.0
26	2	7	65.1
40	L/O	2	60.0
42	3B	2	60.0
43	3B	3	57.0
46	L/O	2	63.0
50	4 A/B	4	63.5
56	4C	3	70.4
57	4C	4	69.2

Coal: Illinois No. 6 Crown II Mine
Catalysts: None: Solvent/Coal = 2=1
399°C 13.0° MPa of nitrogen atmosphere

TABLE 6.7

Inspections of Naphtha Stabilizer Bottoms

-Distribution and Elemental Analysis

Period No.	API	Distribution, W% IBP-177C W%	177-288C W%	288-343C W%	343C+ W%	Elemental Analysis Carbon W%	Hydrogen W%	Nitrogen wppm	Sulfur wppm
4	29.6	15.6	28.5	35.6	20.3	86.47	12.03	273	491
9	30.3	19.7	32.5	32.6	15.2	87.90	12.66	257	172
17	32.6	24.8	37.1	35.4	2.7	87.14	12.45	386	121
19	32.0	22.2	40.1	29.4	8.3	86.95	12.55	394	139
22	33.0	24.9	42.7	27.2	5.2	86.97	12.61	306	116
24	33.3	25.5	43.7	25.6	5.2	87.02	12.51	416	120
26	32.8	25.1	43.9	26.2	4.8	86.86	12.55	352	126
43	32.5	23.1	41.5	29.4	6.0	86.37	12.36	581	345
49	32.5	25.7	46.5	23.1	4.8	86.30	12.29	836	323
56	30.1	24.1	44.3	25.6	6.0	86.46	11.78	1635	483
57	30.3	24.5	44.5	25.6	5.5	86.70	11.87	1419	324
End-Use Sample (Trailer)									
Front	32.9	23.8	43.8	26.3	6.2	86.31	12.36	549	264
	32.7	23.9	44.7	27.0	4.4	86.95	12.43	582	329
Middle	32.7	27.8	43.2	23.7	5.3	86.96	12.38	717	330
Rear	31.8	27.2	45.9	22.4	4.5	86.95	12.08	1225	507

TABLE 6.8

Inspections of Naphtha Stabilizer Bottoms

-Nitrogen and Sulfur Content of Sub-Fractions

Period No.	Nitrogen Content [wppm] IBP-177C	177-288C	288-343	343C+
4	52	289	345	406
9	78	340	404	494
19	139	452	492	565
26	106	429	416	441
43	239	761	569	871
57	626	1778	1722	1978
Tralier (front)	175	651	612	706

Period No.	Sulfur Content [wppm] IBP-177C	177-288C	288-343	343C+
4	98	133	369	461
9	64	82	98	157
19	124	156	137	160
26	120	136	104	134
43		394	221	252
57	310	519	374	444
Tralier (front)	144	264	168	289

TABLE 6.9

Inspection of Sour Water

Period No.	Concentration, wt%	
	Nitrogen	Sulfur
1	2.99	3.58
2	3.36	2.40
3	3.87	1.95
4	3.99	1.77
6	1.79	2.35
7	2.65	1.33
8	2.63	1.12
9	2.72	1.32
19	1.97	1.21
22	2.11	2.65
24	1.99	1.22
26	2.09	1.31
43	1.21	1.26
49	1.02	0.94
50	1.12	0.71
54	0.66	0.86
57	1.82	1.06

TABLE 6.10

Sour Water Sample Analysis (Period 44)

Inspection	Units	260-04-44	Limits
pH		8.67	6-10
Chloride	MG/L	222	
Nitrogen, Ammonia	MG/L	15,900	100
Sulfide, Total	MG/L	114,000	
Total Organic Carbon (TOC)	MG/L	2,530	
Phenols	MG/L	1,290	1
Total Suspended Solids (TSS)	MG/L	41	150
Biological Oxygen Demand (BOD)	MG/L	15,200	150
Chemical Oxygen Demand (COD)	MG/L	97,000	150

Note: Analyses performed by Environmental Science and Engineering, Inc. Plymouth, PA

TABLE 6.11

Analyses of Reactor Liquid Flash Drum Bottoms (0-46)

Condition	1		2		3B	4b	4c	
Periods	17	19	24	26	43	49	56	57
Recycle Mode	Ashy	Ashy	Solid-free	Solid-free	Solid-free	Solid-free	Solid-free	Solid-free
Solv./Coal	1.2	1.2	1.2	1.2	1.2	1.0	0.9	0.9
Composition [W%]								
IBP-524°C	59.15	69.00	72.14	72.99	57.72	60.01	57.01	59.38
524°C+	15.54	14.22	14.22	13.99	22.96	25.24	28.07	24.53
Unreac. Coal	4.10	4.60	4.00	3.94	5.65	4.64	4.42	4.48
Ash	11.21	12.18	9.57	9.08	13.68	10.12	10.5	11.61
Elemental Analysis [W%]								
Carbon	77.78	77.33	79.69	79.62	74.96	78.8	78.76	78.33
Hydrogen	8.70	8.65	9.04	8.86	7.93	8.12	7.11	7.26
Nitrogen	0.212	0.262	0.282	0.26	0.384	0.487	0.606	0.552
Sulfur	1.002	0.506	0.735	0.734	0.770	0.974	1.028	1.09
H/C Ratio	1.34	1.34	1.36	1.34	1.27	1.24	1.08	1.11

TABLE 6.12

Inspection of Interstage Samples

Sample Period	Interstage 40A	Interstage 42B	O-46 26	O-46 49
Pressure Filtration, W%				
Liquid	42.01	50.70	73.12	62.66
Solid	57.99	49.30	26.88	37.34
Pressure Filter Liquid				
API	13.0	12.6	10.2	n/a
IBP [C]	252	218	142	138
Distribution [W% Solid]				
IBP-343C	5.54	10.69	6.73	5.56
343-454C	19.22	24.62	40.65	31.64
454-524C	7.04	6.76	14.04	10.14
524C+	10.21	8.62	11.7	15.32
Elemental Analysis [W% Liquid]				
Carbon	88.13	88.30	88.47	88.54
Hydrogen	10.81	10.65	10.17	9.62
Nitrogen	0.21	0.24	0.23	0.39
Sulfur	0.09	0.28	0.07	0.243
Oxygen [By Diff.]	0.76	0.53	1.06	1.21
H/C Ratio	1.47	1.45	1.38	1.30
Pressure Filter Solid				
ASTM Ash [W% Solid]	12.63	11.49	9.52	10.21
S in Ash [W% Ash]	1.58	2.11		
SO3-Free ASTM Ash [W% Solid]	12.13	10.88	9.23	9.90
Distribution [W% Solid]				
IBP-524C	24.41	20.47	11.28	12.31
524C+ Resid	11.30	9.63	10.15	19.98
Unreacted Coal	10.14	8.31	3.79	4.86
Ash (SO3-free)	12.13	10.88	9.23	9.90
Coal Conversion [W%]	88.74	90.77	96.3	95.9
Elemental Analysis [W% Solid]				
Carbon	65.22	64.13	55.56	62.46
Hydrogen	6.44	6.29	5.28	5.61
Nitrogen	0.64	0.69	0.34	0.65
Sulfur	2.61	2.84	2.54	2.20
Ash (SO3-free)	12.13	10.88	9.23	9.90
Oxygen [By Diff.]	12.96	15.17	27.05	19.18
H/C Ratio	1.18	1.18	1.14	1.08

TABLE 6.13a

Analysis of True Boiling Point Fractions From Naphtha Stablizer Bottoms

Run POC-01 Period: 15/17

Fraction	Whole	IBP-177C	177-288C	288-343C	343C+
API Gravity	32.6	51.5	28.9	20.5	18.3
Elemental [W%]					
Carbon	87.15	85.13	87.26	84.42	87.79
Hydrogen	12.69	14	12.41	11.63	11.51
Oxygen (Direct)	<0.02	<0.02	<0.02	<0.02	<0.02
Sulfur [ppm]	214.4	43	116.9	110.2	226
Nitrogen, ppm (Antek)	92.9	42	244.5	303.2	363.2
Flash Point [C]		< -6.7	80.0	154.4	
Anline Point [C]			36.7	44.7	
Pour Point [C]				-7.6	3.0
Smoke Point [mm]			14.1	11.3	
Bromine No. [g/100g]		NES	4.75	3.33	
Cetane Index			36.2	34.6	
Octane No.		62.0			
Octane No. (Research)		66.6			
Refractive Index		1.4279	1.4844	1.5188	
Heating Value [1xE+07 Joule/Kg	4.40	4.37	4.45	4.44	
CCR [W%]	0			0	0.12
Molecular Weight	286	217	224	274	312
Viscosity CST @26.7C	2.3	0.81		14.17	NES
@37.8C	1.85	0.71			
Solubility [W%]					
Pentane Insoluble	0.13				0.36
Toluene Insoluble	0.08				0.21
Quinoline Insoluble	0.17				0.19

TABLE 6.13b

Analysis of True Boiling Point Fractions From Naphtha Stablizer Bottoms

POC-01 Period: 24/26

Fraction	Whole	IBP-177C	177-288C	288-343C	343C+
API Gravity	33.4	51.4	27.7	20.6	16.3
Elemental [W%]					
Carbon	87.4	85.54	87.9	88.3	88.1
Hydrogen	12.75	14.05	12.55	11.97	11.28
Oxygen (Direct)	<0.02	<0.02	<0.02	<0.02	<0.02
Sulfur, ppm	239.8	40.8	77.5	93.8	652.8
Nitrogen, ppm (Antek)	152.6	88.3	146	187	262.8
Flash Point [C]		< -6.7	84.4	157.2	
Anline Point [C]			34.2	44.2	
Pour Point [C]				-52.8	NES
Smoke Point [mm]			13.4	10.6	
Bromine No. (g/100g)		7.49	4.39	4.19	
Cetane Index			33.8	34.8	
Refractive Index		1.4294	1.4889	1.5146	
Heating Value [Joule/Kg]					
CCR [W%]	0			0	0.82
Molecular Weight	234	215	230	264	329
Viscosity CST @ 26.7C	2.12	0.81		15.02	NES
@ 37.8C	1.76	0.71		9.64	NES
Solubility [W%]					
Pentane Insoluble	0.043				3.09
Toluene Insoluble	0				0.43
Quinoline Insoluble	0				0.11

TABLE 6.13c

Analysis of True Boiling Point Fractions From Naphtha Stablizer Bottoms

Run POC-01 Period: 48/49/50

Fraction	Whole	IBP-177C	177-288C	288-343C	343C+
API Gravity	32.8	49.5	27.4	20.3	13.5
Elemental [W%]					
Carbon	87.51	85.19	87.99	88.48	87.8
Hydrogen	12.66	13.74	12.27	11.76	10.71
Oxygen [Direct]	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
Sulfur, ppm	310	252.6	440.7	403.2	1349
Nitrogen [ppm] Antek	114.7	113.9	290.3	340.8	537
Flash Point [C]		< -6.7	81.1	157.2	
Anline Point [C]			29.2	39.7	
Pour Point [C]				-48.0	NES
Smoke Point [mm]			11.9	8.8	
Bromine No. (g/100g)		11.16	6.65	4.93	
Cetane Index			34.2	34.4	
Octane No.		63.2			
Octane No. (Research)		68.3			
Refractive Index		1.432	1.4907	1.1517	
Heating Value [1xE+07 Joule/Kg]	4.37	4.42	4.42	4.39	
CCR [W%]	0				0.13
Molecular Weight	259	200	206	245	325
Viscosity CST @26.7C	2.04	0.84		13.05	NES
@37.8C	1.67	0.74		8.48	NES
Solubility [W%]					
Pentane Insoluble	0.03				4.33
Toluene Insoluble	0.09				0.23
Quinoline Insoluble	0.05				0.07

TABLE 6.13d

Analysis of True Boiling Point Fractions From Naphtha Stablizer Bottoms

Run POC-01 Period: 56/57

Fraction	Whole	IBP-177C	177-288C	288-343C	343C+
API Gravity	30.8	50.8	25.2	17.5	9.4
Elemental [W%]					
Carbon	87.02	85.32	87.67	88.37	88.52
Hydrogen	11.98	13.71	11.74	11	9.63
Sulfur, ppm	629	386.4	459.6	578.4	1721.8
Nitrogen, ppm (Antek)	1235.2	333.4	1482.4	1664.8	2788.1
Flash Point [C]		< -6.7	81.1	NES	
Anline Point [C]			18.9	24.4	
Pour Point [C]				-46.0	NES
Smoke Point [mm]			10.9	8.3	
Bromine No. (g/100g)		15.35	15.12	8.31	
Cetane Index			30.4	31.3	
Refractive Index		1.4312	1.2988	1.5356	
Heating Value [1xE+07]Joule/Kg					
CCR [W%]	0			0	1.77
Molecular Weight	2.7	204	218	259	317
Viscosity CST @ 27.6C	2.22	0.79		16.26	NES
@ 37.8C	1.8	0.7		10.11	NES
Solubility [W%]					
Pentane Insoluble	0.13				4
Toluene Insoluble	0.36				1.32
Quinoline Insoluble	0.01				1.13

TABLE 6.14a

Analysis of True Boiling Point Fractions
Simulated Distillation
Period: 15/17

Cumulative Volume	Temperature [C]				
	Whole	IBP-177C	177-288C	288-343C	343C+
0.5	82	79	189	259	316
5	121	108	203	294	351
10	143	114	212	300	356
15	170	116	219	304	359
20	190	122	225	307	362
25	204	129	231	310	364
30	219	131	238	313	367
35	233	133	243	316	369
40	245	139	248	319	372
45	258	146	255	322	374
50	268	156	259	324	376
55	275	160	264	327	379
60	284	166	268	329	382
65	294	172	269	332	384
70	304	179	274	335	388
75	313	183	278	338	391
80	323	188	282	341	396
85	334	192	287	344	401
90	347	196	292	348	409
95	367	199	297	354	422
100	386	203	302	359	436

TABLE 6.14b

Analysis of True Boiling Point Fractions
Simulated Distillation
Period: 24/26

Cumulative	Temperature [C]				
V %	Whole	IBP-177C	177-288C	288-343C	343C+
1	49	51	168	253	318
5	91	81	188	293	358
10	112	87	196	299	363
15	137	89	204	304	367
20	163	93	213	308	370
25	183	104	222	311	372
30	196	106	229	314	374
35	210	108	236	318	376
40	226	113	242	321	378
45	238	120	249	334	379
50	249	127	257	326	381
55	261	133	261	328	383
60	270	139	266	331	386
65	277	147	271	334	388
70	286	156	273	337	390
75	296	160	278	341	392
80	306	166	283	345	396
85	316	171	289	349	400
90	329	180	295	355	407
95	347	194	304	361	413
99.5	364	207	313	367	426

TABLE 6.14c

Analysis of True Boiling Point Fractions
Simulated Distillation
Period: 48/49/50

Cumulative	Whole	IBP-177C	177-288C	288-343C	343C+
V %	Temperature [c]				
1	57	60	126	270	323
5	104	90	191	290	358
10	126	95	202	297	363
15	153	102	211	302	367
20	178	111	219	306	369
25	193	113	227	309	372
30	208	115	234	312	373
35	222	120	240	315	376
40	235	128	247	319	377
45	246	134	252	322	379
50	258	142	260	324	381
55	267	147	264	328	383
60	273	156	268	331	386
65	281	163	271	334	388
70	289	168	276	337	391
75	299	176	281	342	395
80	308	182	286	347	399
85	319	189	292	353	407
90	333	196	300	360	421
95	359	202	309	368	441
99.5	383	210	318	376	456

TABLE 6.14d

Analysis of True Boiling Point Fractions
Simulated Distillation
Period: 56/57

Cumulative	Whole	IBP-177C	177-288C	288-343C	343C+
V Percent	Temperature [C]				
1	56	53	164	273	318
5	102	83	189	296	356
10	123	89	197	302	363
15	153	91	207	307	367
20	177	96	213	310	370
25	193	107	222	313	373
30	207	109	229	316	374
35	222	111	236	319	377
40	234	115	242	322	379
45	246	122	248	323	382
50	258	127	257	326	384
55	267	136	261	328	387
60	273	139	267	331	389
65	281	147	269	333	392
70	289	154	274	336	396
75	298	161	280	338	400
80	306	166	284	342	405
85	317	172	291	346	412
90	330	181	297	349	423
95	343	189	307	353	434
99.5	357	197	316	356	441

TABLE 6.15

ROSE-SR^{SM} Performance: Off-line Tests (11/20-12/1/93)

	Test I				Test II			
	Feed	Product			Feed	Product		
		DAO	Bottom	Total		DAO	Bottom	Total
Amount [Kg]	304	139	178	317	292	218	94	312
Recovery [Wt%]				104				107
Analysis [Wt%]								
IBP-454°C	21.33	35.28				50.24		
454-524°C	13.72	21.63				16.49		
524°C+	34.77	20.31	33.33			17.11	19.2	
Unreacted Coal	8.99	7.67	19.50		10.09	6.63	32.65	
Ash	21.20	15.11	47.17		14.12	9.53	48.15	
Mass Balance [Kg]								
IBP-454°C	64.8	48.9		48.9		109.6		109.6
454-524°C	41.6	30.0		30.0		36.0		36.0
524°C+	105.5	28.1	59.2	87.3		37.3	18.1	55.4
Unreacted Coal	27.3	10.6	34.6	45.2	29.5	14.4	30.9	45.3
Ash	64.4	21.0	83.8	104.8	41.3	20.8	45.5	66.3

TABLE 6.16

ROSE-SR^{SM} UNIT PERFORMANCE
Ash and Overall Material Balance

Period No.	Feed	Product DAO	Product Bottoms	Total	Overall Recovery	Ash Recovery
	Mass Balance [lb/h]					
14	62.20	18.90	43.87	62.77	100.92	89.16
15	64.90	23.00	41.38	64.38	99.20	123.05
17	76.80	37.60	38.00	75.60	98.44	64.89
19	57.10	16.00	42.20	58.20	101.93	99.79
21	45.10	19.30	30.50	49.80	110.42	141.19
22	89.60	42.40	47.00	89.40	99.78	94.35
24	72.30	39.00	37.10	76.10	105.26	110.35
26	79.00	41.10	39.00	80.10	101.39	105.38
28	112.30	60.80	51.20	112.00	99.73	n/a
43	77.40	32.40	36.70	69.10	89.28	90.22
46	65.60	26.70	37.10	63.80	97.26	52.63
47	88.20	33.80	60.30	94.10	106.69	98.13
48	94.30	33.20	64.60	97.80	103.71	129.75
49	82.20	35.50	48.20	83.70	101.82	101.19
50	128.60	56.80	77.00	133.80	104.04	105.66
54	52.00	20.10	32.50	52.60	101.15	75.45
56	125.60	44.00	86.80	130.80	104.14	109.96
57	144.90	50.10	96.30	146.40	101.04	99.57

TABLE 6.17

POC-01 ROSE-SR^{SM} UNIT PERFORMANCE

TGA Simulated Distillation

	Feed (O-61)				DAO (O-65)				Bottoms (O-63)		
	IBP-524C	524C+	Ash		IBP-524C	524C+	Ash		IBP-524C	524C+	Ash
14				14				14	32.10	42.91	24.99
15	23.56	50.13	26.31	15				15	8.97	44.22	46.81
17				17*	53.16	35.85	10.99	17	37.78	44.61	17.61
19				19*	63.30	33.06	3.64	19	17.46	47.77	34.77
21	36.70	41.69	21.61	21*	52.30	39.20	8.50	21	13.90	46.45	39.65
22				22				22	11.55	48.61	39.84
24				24*	63.42	35.71	0.87	24	7.83	46.33	45.84
26				26*	64.71	35.04	0.25	26	9.14	46.48	44.38
29	30.65	51.07	18.28	29				29			
40	12.51	63.84	23.65	40				40			
42	23.68	52.13	24.19	43*	63.44	36.03	0.53	43	6.06	53.40	40.54
46	23.20	52.22	24.58	46				46	18.11	58.97	22.92
47	28.15	48.60	23.25	47				47	17.67	48.95	33.38
48	28.14	52.60	19.26	48				48	6.07	58.65	35.28
49				49*	58.90	40.28	0.82	49	5.49	61.01	33.50
50				50				50	6.82	63.62	29.56
54	18.68	57.44	23.88	54				54			
56				56*	63.75	35.92	0.33	56	9.30	61.96	28.74
57				57*	60.20	39.13	0.67	57	9.69	62.90	27.41

Note:
1. DI160 Distillation Data
2. 524C+ Fraction contains unreacted coal

TABLE 6.18

Compound Class Type Analysis of Rose-SR^{SM} Feed and Products

Analysis of ROSE Feed (O-61), W%

	Pentane Soluble	Asphaltene	Preasphaltene	IOM	Ash
14	55.52	6.99	1.33	10.83	25.33
15	53.92	7.65	3.32	10.38	24.73
17	59.80	8.79	1.05	8.90	21.46
19	45.27	15.27	2.14	10.50	26.82
21	56.17	11.09	1.63	9.94	21.17
22	56.60	10.22	1.56	9.57	22.05
24	56.19	12.11	0.99	9.01	21.70
26	53.95	14.78	5.94	4.60	20.73
29	56.76	14.06	3.75	7.50	17.93
43	38.79	25.51	5.84	8.76	21.10
46	51.91	14.17	-1.33	10.49	24.76
47	44.48	18.74	5.07	8.37	23.34
48	46.31	21.27	5.18	8.26	18.98
49	43.27	24.00	5.16	8.01	19.56
50	37.14	29.82	9.09	7.92	16.03
54	40.75	21.78	5.40	8.50	23.57
56	35.08	29.71	9.81	7.44	17.96
57	33.60	30.32	10.20	8.10	17.78

Analysis of ROSE Bottoms (O-63), W%

	Pentane Soluble	Asphaltene	Preasphaltene	IOM	Ash
14			0.42	11.54	25.65
15			7.23	19.69	44.76
17			1.48	7.92	17.27
19	36.72	12.88	2.81	12.77	34.83
21			5.30	15.99	38.82
22			3.83	16.64	39.58
24			4.48	19.25	45.75
26	20.42	10.59	6.05	18.70	44.24
29					
43	8.64	24.61	10.22	16.46	40.07
46			5.88	13.35	23.04
47			0.91	12.34	33.50
48			7.49	13.08	35.28
49			11.34	13.64	33.15
50	1.81	42.07	12.73	15.17	28.22
54				13.96	28.30
56			11.50	11.13	28.41
57	4.14	40.94	16.72	11.61	26.59

Analysis of DAO (O-65), W%

	Pentane Soluble	Asphaltene	Preasphaltene	IOM	Ash
14	74.93	2.32	0.19	7.77	14.79
15			-0.02	4.06	5.34
17	79.70	3.42	0.49	5.40	10.99
19	91.70	2.32	0.04	2.29	3.65
21			-0.39	6.39	8.50
22	97.41	2.14	-0.25	0.61	0.09
24	95.07	3.40	-0.61	1.27	0.87
26	96.23	3.33	0.00	0.43	0.01
29					
43	94.91	4.87	0.00	0.13	0.09
46					
47					
48			-0.17	1.11	1.30
49	92.34	6.08	-0.40	1.16	0.82
50	92.15	6.60	0.00	1.16	0.09
54			-0.56	0.89	0.25
56	96.06	3.34	-0.32	0.59	0.33
57	92.03	6.94	0.00	0.94	0.09

TABLE 6.19

PDU-260 ROSE-SR^{SM} SECTION

Performance On W% MAF Basis

Period	04	09	14*	17*	19	24*	26	43*	49*	56*	57*
Solids Separation	Vacuum Still		ROSE-SR^{SM}								
Organic Rejection	48.5	36.5	24.3	23.2	20.3	14.2	15.8	12.6	17.5	30.0	34.4
Energy Rejection			23.1	19.8	22.1	15.5	17.2	12.5	18.6	30.0	33.5
Coal Conversion	92.1	95.6	94.6	94.5	95.9	95.2	95.2	95.3	95.3	95.9	95.4
Rejection of 975°F-	29.8	19.9	10.5	10.6	5.4	2.0	n/a	n/a	n/a	3.6	4.5

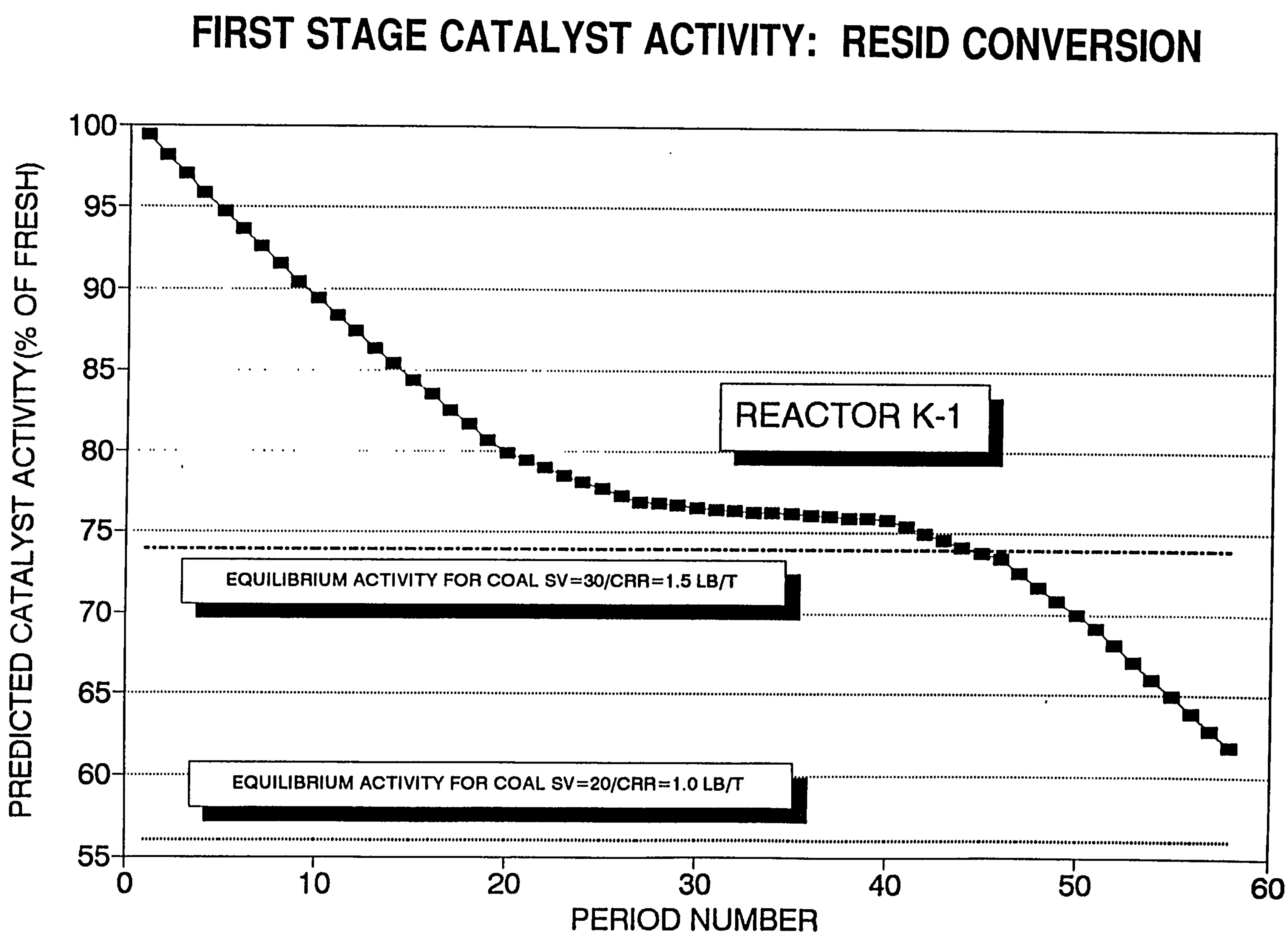
Energy Rejection is estimated using Dulong Formula

TABLE 6.20

ANALYSIS OF CORE, ROSE-SR^{SM} BOTTOM SAMPLES TAKEN FROM PERIOD 19 AND 26

	Period 19	Period 26
Proximate Analysis [Wt%]		
Moisture	0.18	0.29
Volatile Matter (dry)	47.85	33.72
Fixed Carbon (dry)	18.23	22.66
Ash (dry)	33.92	43.63
Elemental Analysis [Wt%]		
Carbon	56.70	47.05
Hydrogen	4.76	3.22
Nitrogen	0.43	0.54
Sulfur	2.81	3.30
TGA Analysis [Wt%]		
IBP-524°C	17.46	9.14
524°C+ Resid	34.94	27.92
Unreacted Coal	12.77	18.70
Ash	34.83	44.24
Molybdenum [wppm]	39	135
Heating Value [x 10^6 Joules/Kg]		
Measured	24.68	19.89
Calculated	25.70	20.15
Sieve Analysis [Wt%]		
Retained on 40 mesh		58.0
Retained on 60 mesh		13.0
Retained on 100 mesh		7.1
Retained on 140 mesh		5.9
Retained on 200 mesh		4.3
Fines		10.3
Loss		1.4
Heating value of Coal [x 10^6 Joules/Kg, dry basis]		
HRI Lab.	29.11	
Core Lab.	29.55	

FIGURE 6.1



FIRST STAGE CATALYST ACTIVITY: RESID CONVERSION
REACTOR K-1
EQUILIBRIUM ACTIVITY FOR COAL SV=30/CRR=1.5 LB/T
EQUILIBRIUM ACTIVITY FOR COAL SV=20/CRR=1.0 LB/T
PREDICTED CATALYST ACTIVITY(% OF FRESH)
100
95
90
85
80
75
70
65
60
55
0
10
20
30
40
50
60
PERIOD NUMBER

FIGURE 6.2

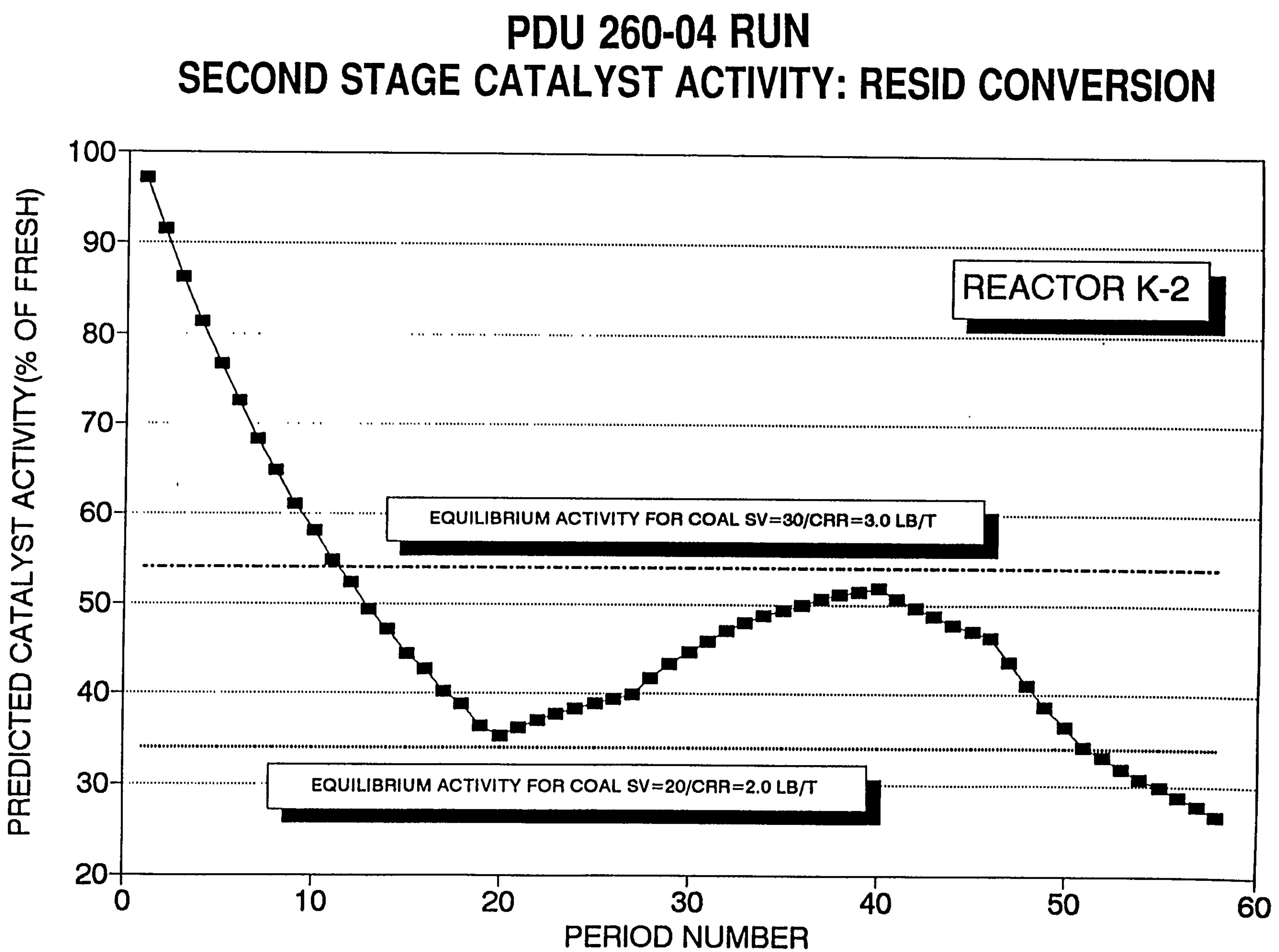
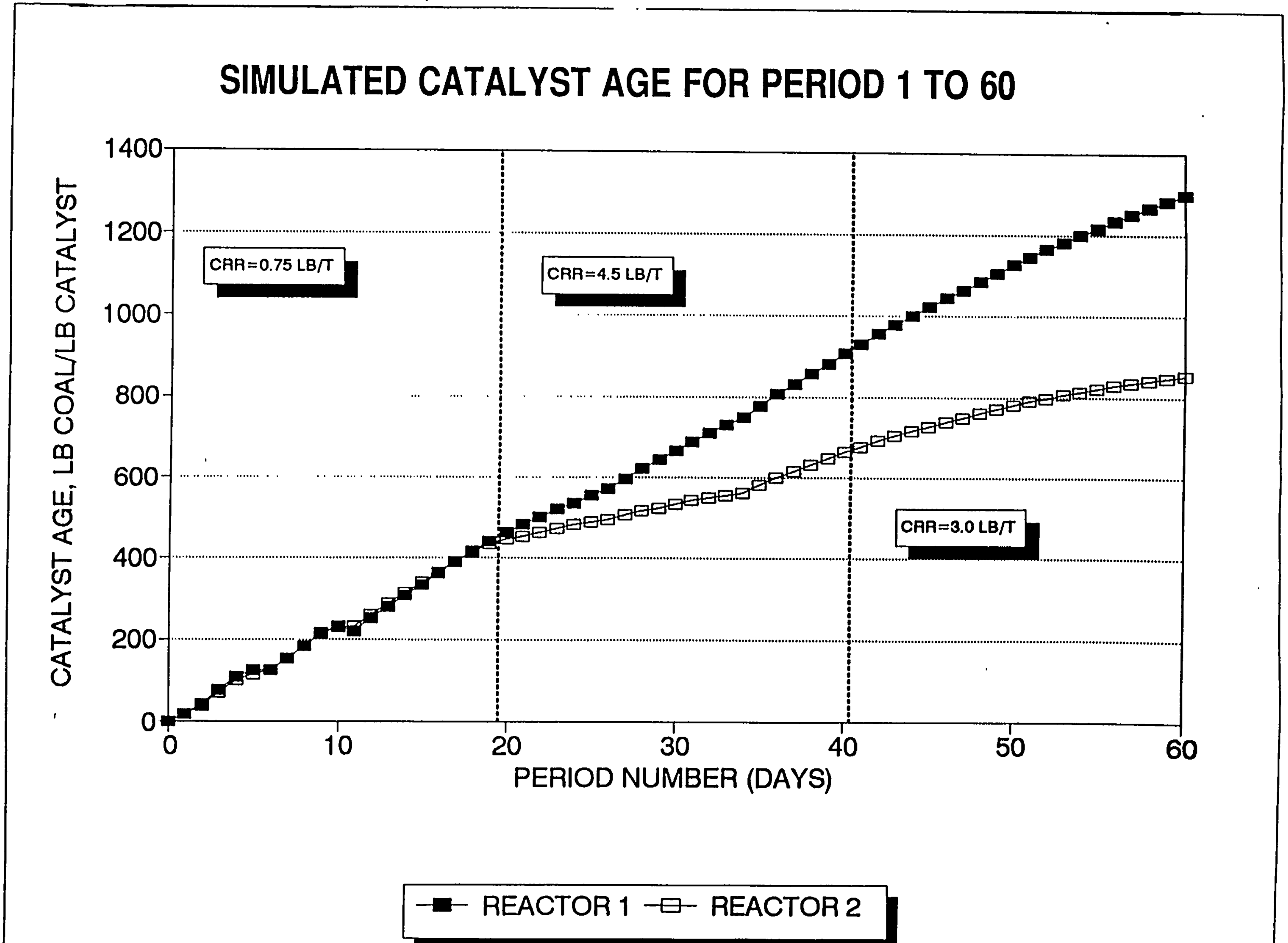
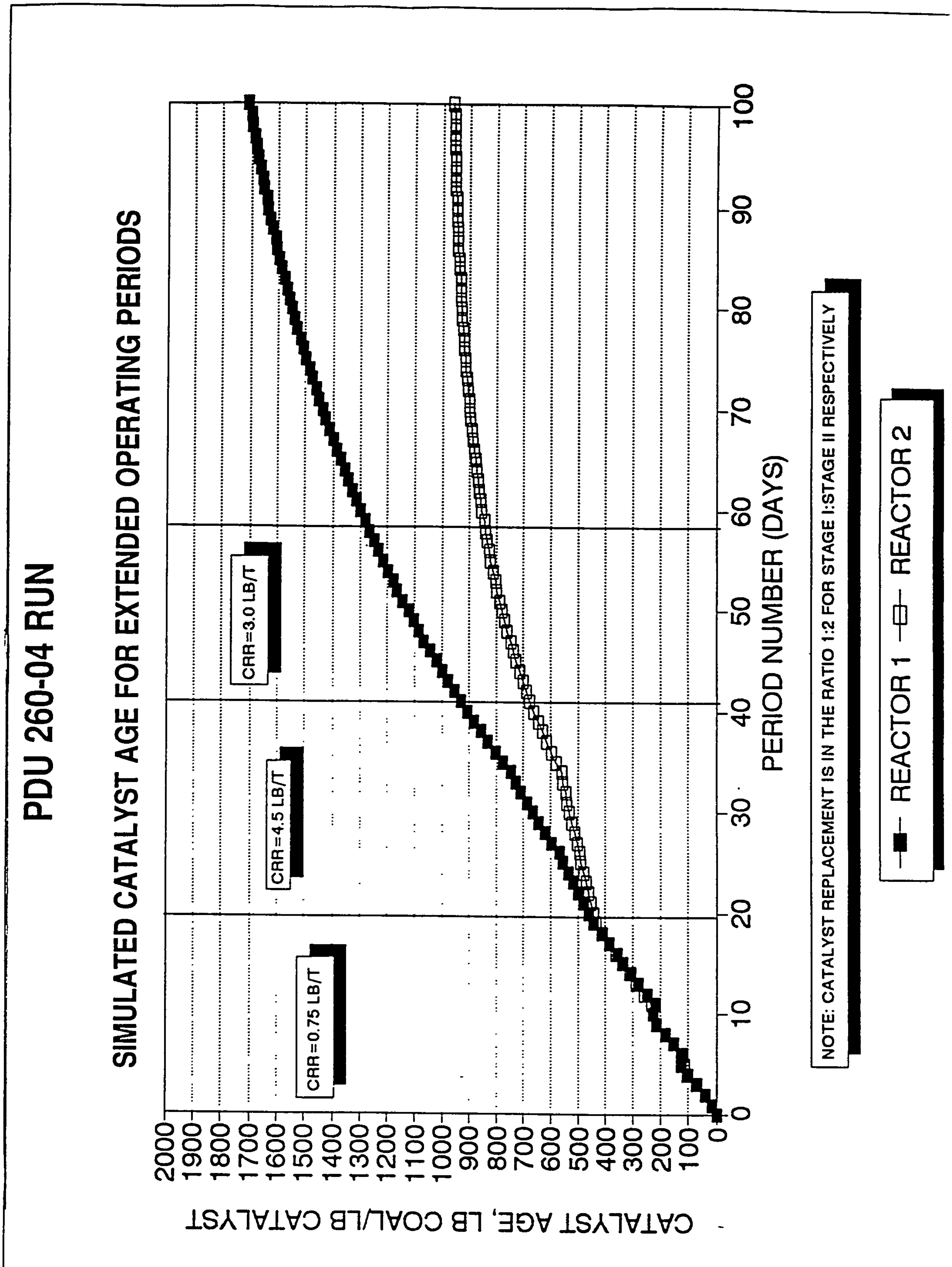

PDU 260-04 RUN
SECOND STAGE CATALYST ACTIVITY: RESID CONVERSION
REACTOR K-2
EQUILIBRIUM ACTIVITY FOR COAL SV=30/CRR=3.0 LB/T
EQUILIBRIUM ACTIVITY FOR COAL SV=20/CRR=2.0 LB/T
PREDICTED CATALYST ACTIVITY(% OF FRESH)
PERIOD NUMBER
100
90
80
70
60
50
40
30
20
0
10
20
30
40
50
60

FIGURE 6.3



SIMULATED CATALYST AGE FOR PERIOD 1 TO 60
CATALYST AGE, LB COAL/LB CATALYST
PERIOD NUMBER (DAYS)
0
200
400
600
800
1000
1200
1400
0
10
20
30
40
50
60
CRR=0.75 LB/T
CRR=4.5 LB/T
CRR=3.0 LB/T
REACTOR 1
REACTOR 2

FIGURE 6.4



PDU 260-04 RUN
SIMULATED CATALYST AGE FOR EXTENDED OPERATING PERIODS
CATALYST AGE, LB COAL/LB CATALYST
PERIOD NUMBER (DAYS)
CRR=0.75 LB/T
CRR=4.5 LB/T
CRR=3.0 LB/T
NOTE: CATALYST REPLACEMENT IS IN THE RATIO 1:2 FOR STAGE I:STAGE II RESPECTIVELY
REACTOR 1
REACTOR 2

FIGURE 6.5

ACTUAL FIRST AND SECOND STAGE CATALYST AGES

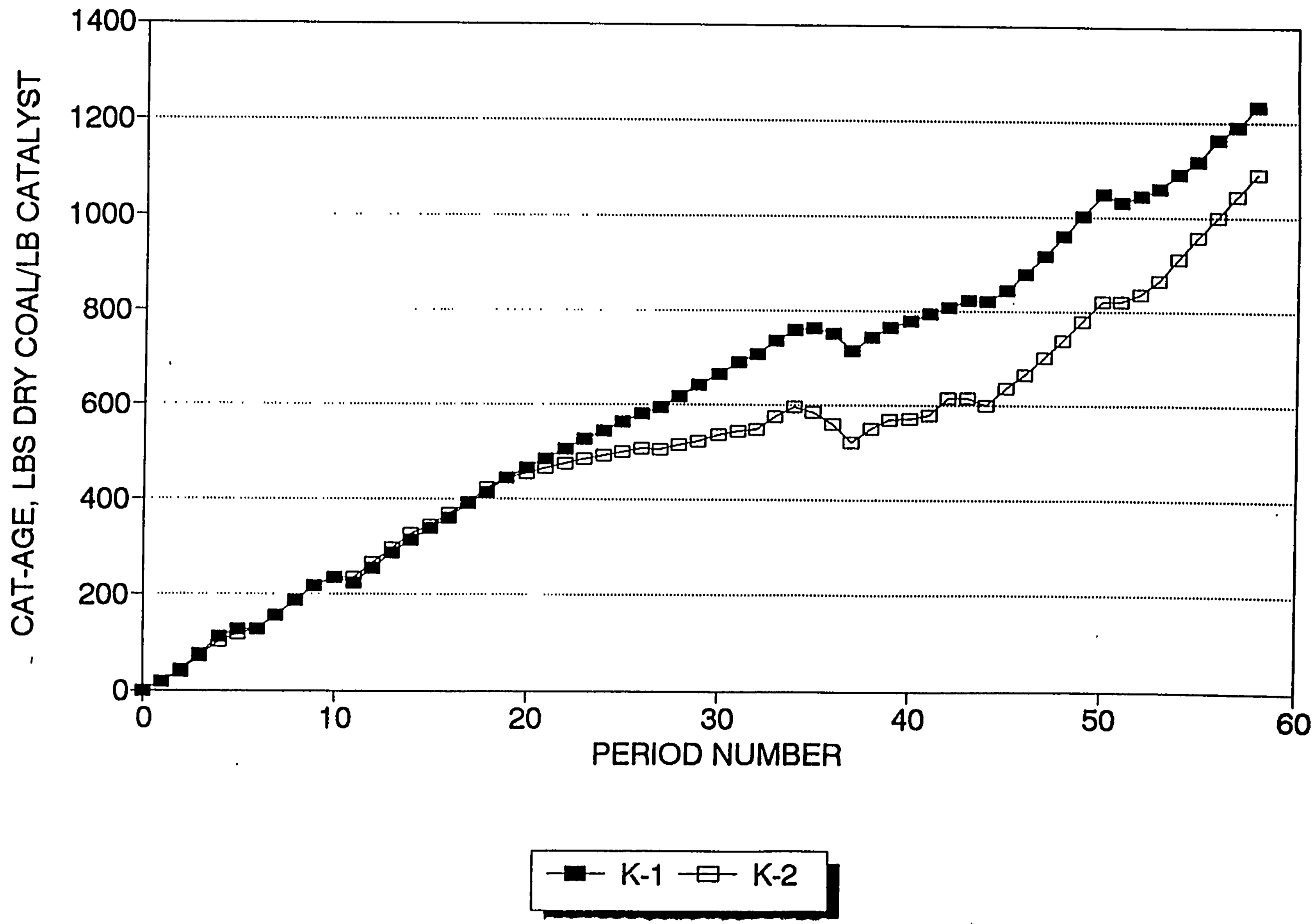
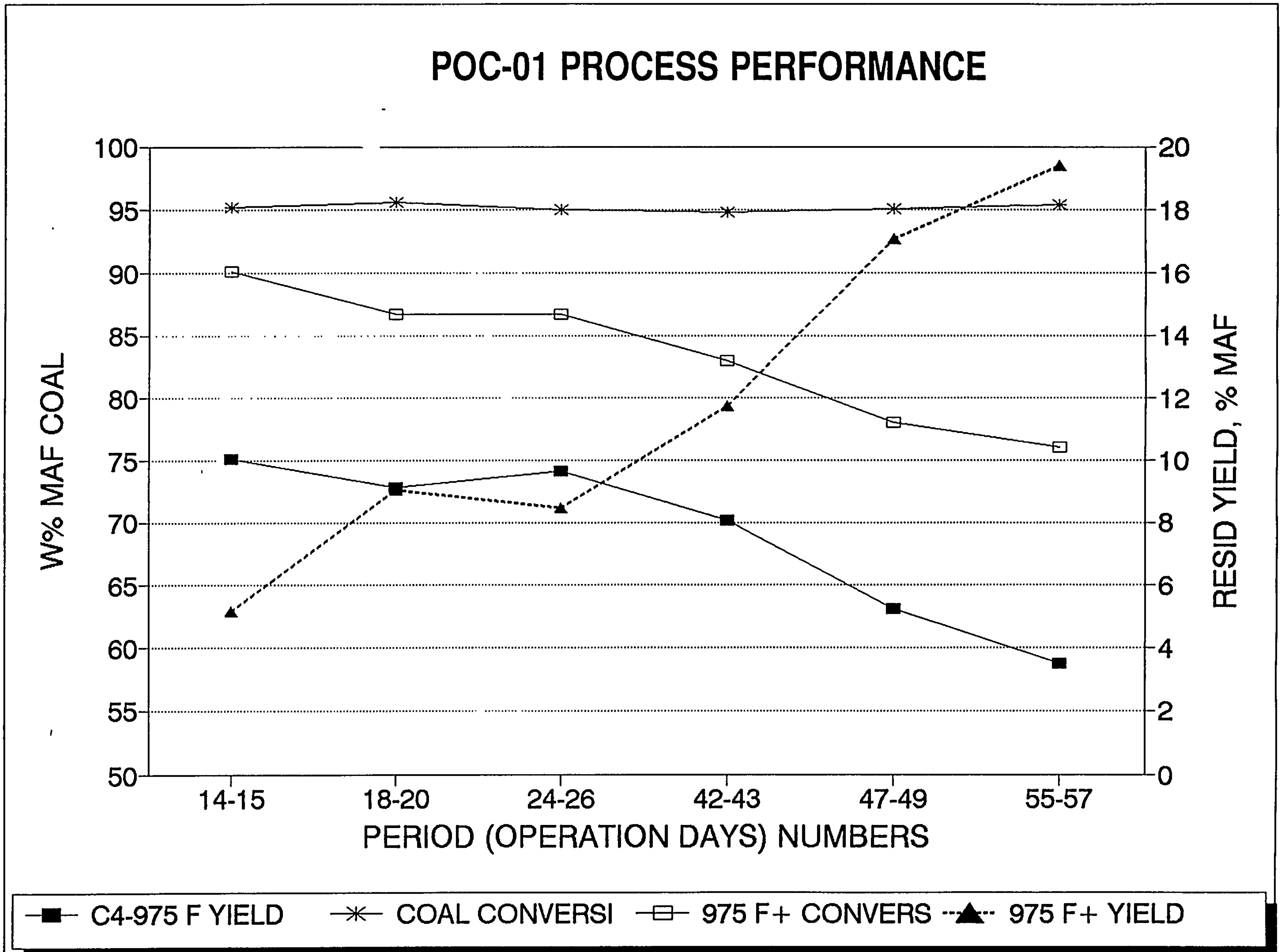


FIGURE 6.6



POC-01 PROCESS PERFORMANCE
W% MAF COAL
RESID YIELD, % MAF
100
95
90
85
80
75
70
65
60
55
50
20
18
16
14
12
10
8
6
4
2
0
14-15
18-20
24-26
42-43
47-49
55-57
PERIOD (OPERATION DAYS) NUMBERS
C4-975 F YIELD
COAL CONVERSI
975 F+ CONVERS
975 F+ YIELD

FIGURE 6.7

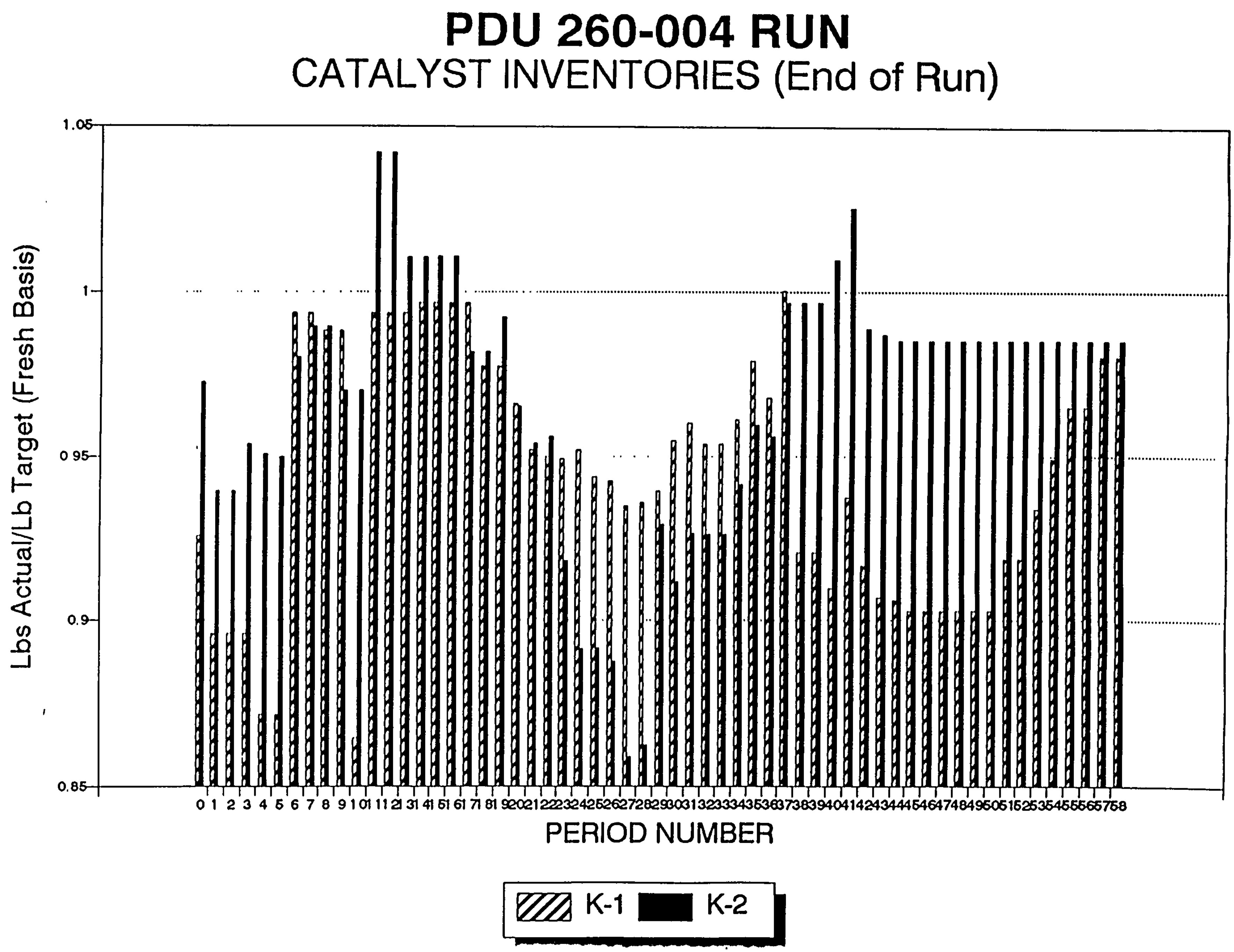

PDU 260-004 RUN
CATALYST INVENTORIES (End of Run)
Lbs Actual/Lb Target (Fresh Basis)
1.05
1
0.95
0.9
0.85
PERIOD NUMBER
K-1
K-2

FIGURE 6.8

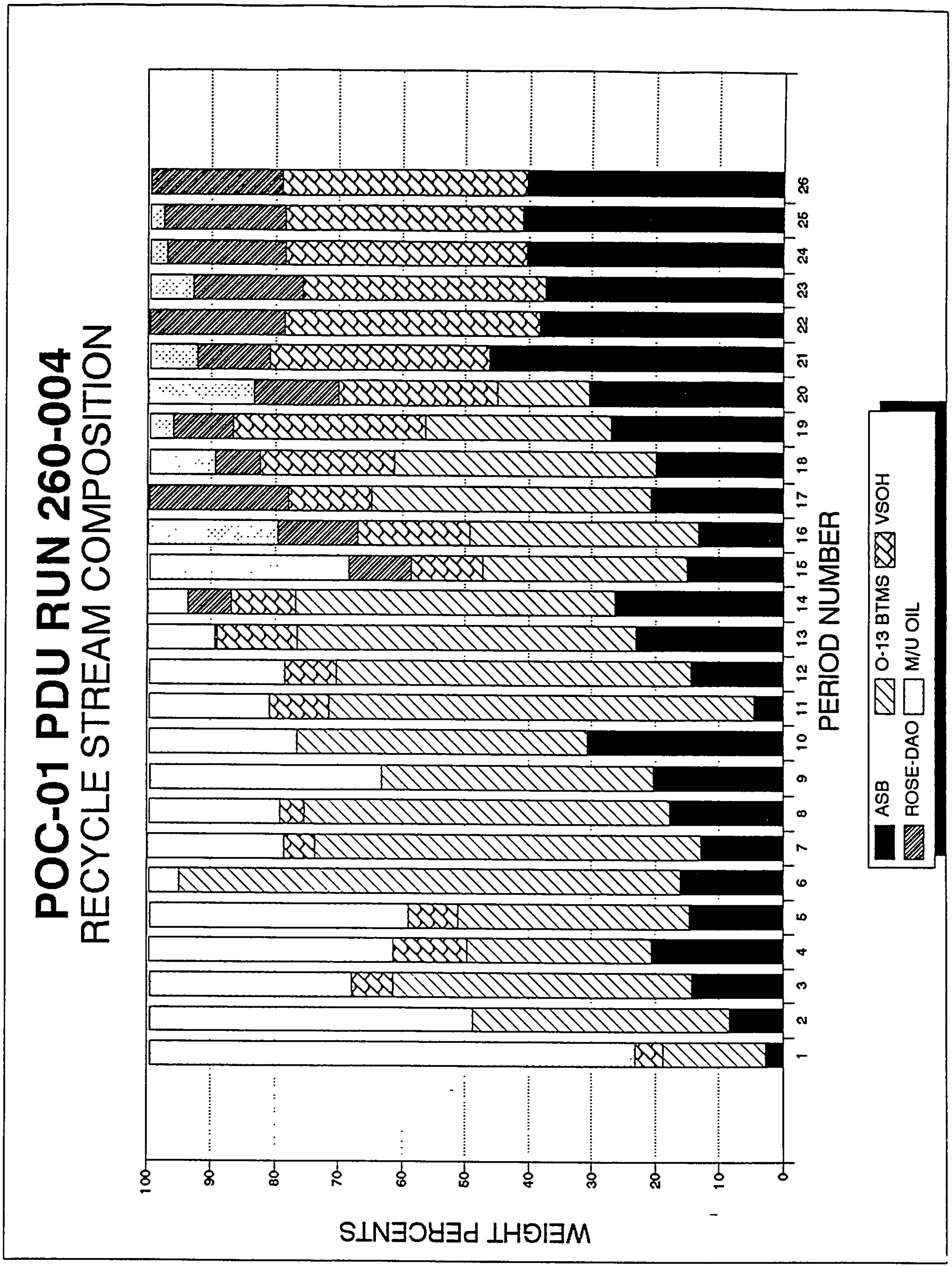
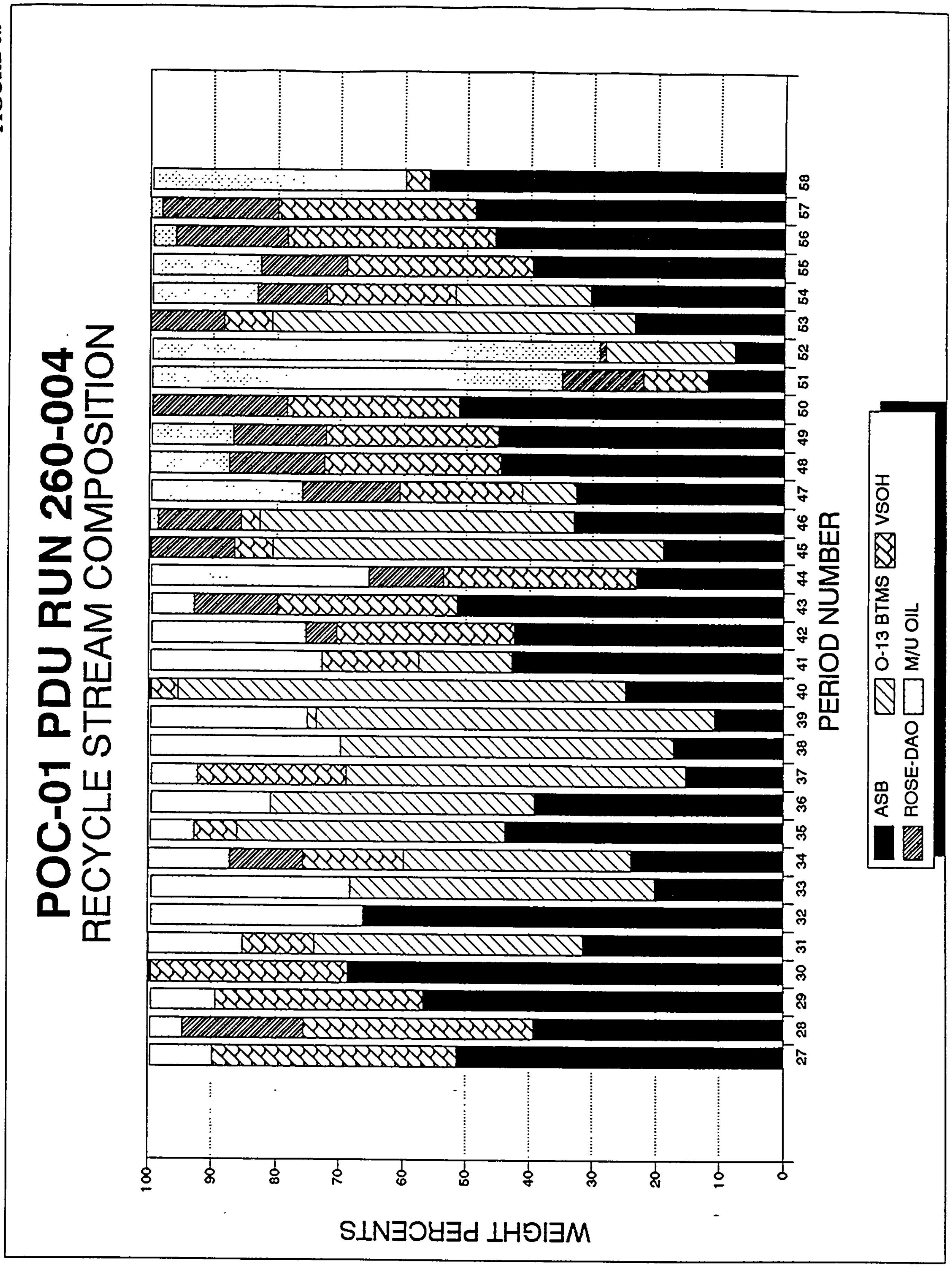
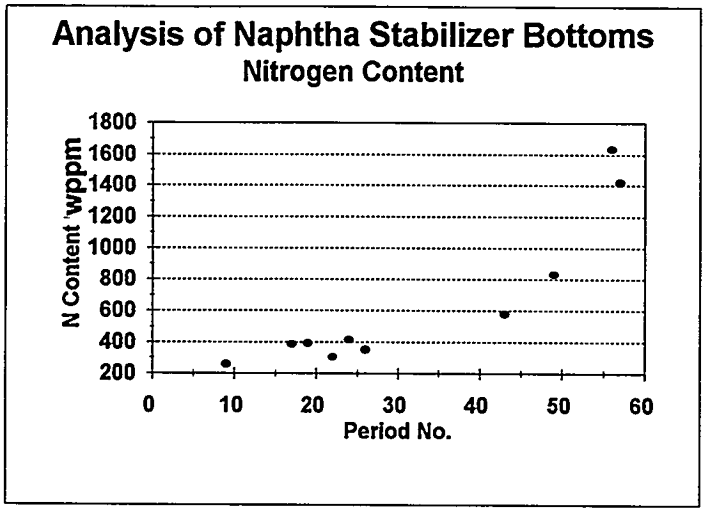

POC-01 PDU RUN 260-004
RECYCLE STREAM COMPOSITION
WEIGHT PERCENTS
0
10
20
30
40
50
60
70
80
90
100
PERIOD NUMBER
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
ASB
O-13 BTMS
VSOH
ROSE-DAO
M/U OIL

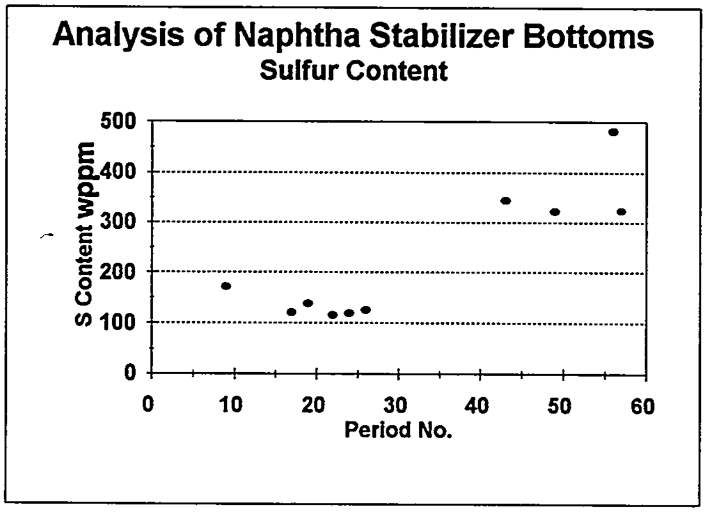
FIGURE 6.9



POC-01 PDU RUN 260-004
RECYCLE STREAM COMPOSITION
WEIGHT PERCENTS
0
10
20
30
40
50
60
70
80
90
100
PERIOD NUMBER
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
ASB
O-13 BTMS
VSOH
ROSE-DAO
M/U OIL

ANALYSIS OF NAPHTHA STABLIZER BOTTOMS





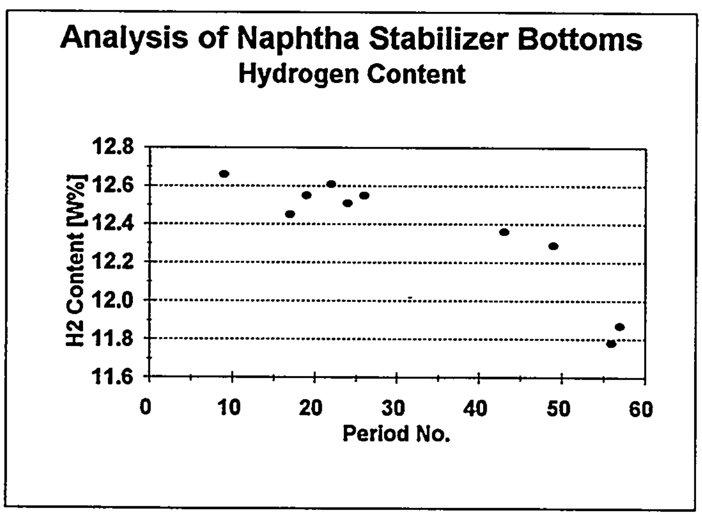
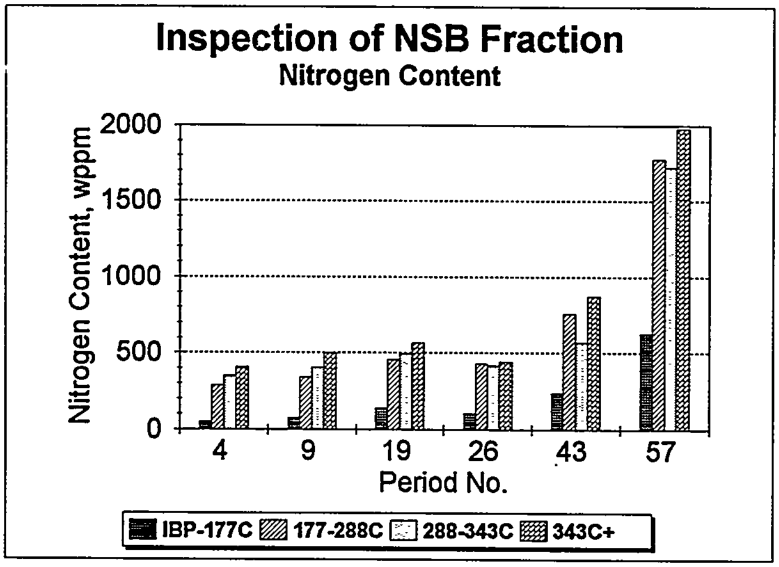


FIGURE 6.11

INSPECTION OF NSB FRACTION
NITROGEN AND SULFUR CONTENT



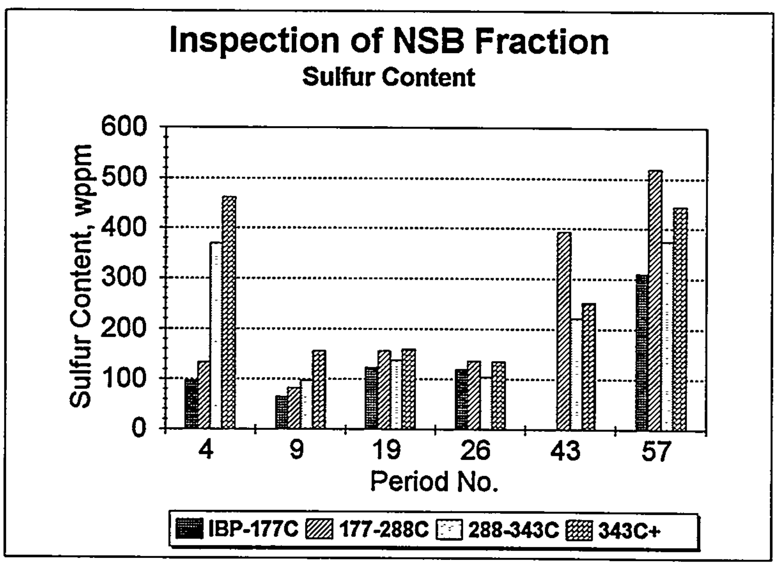


FIGURE 6.12

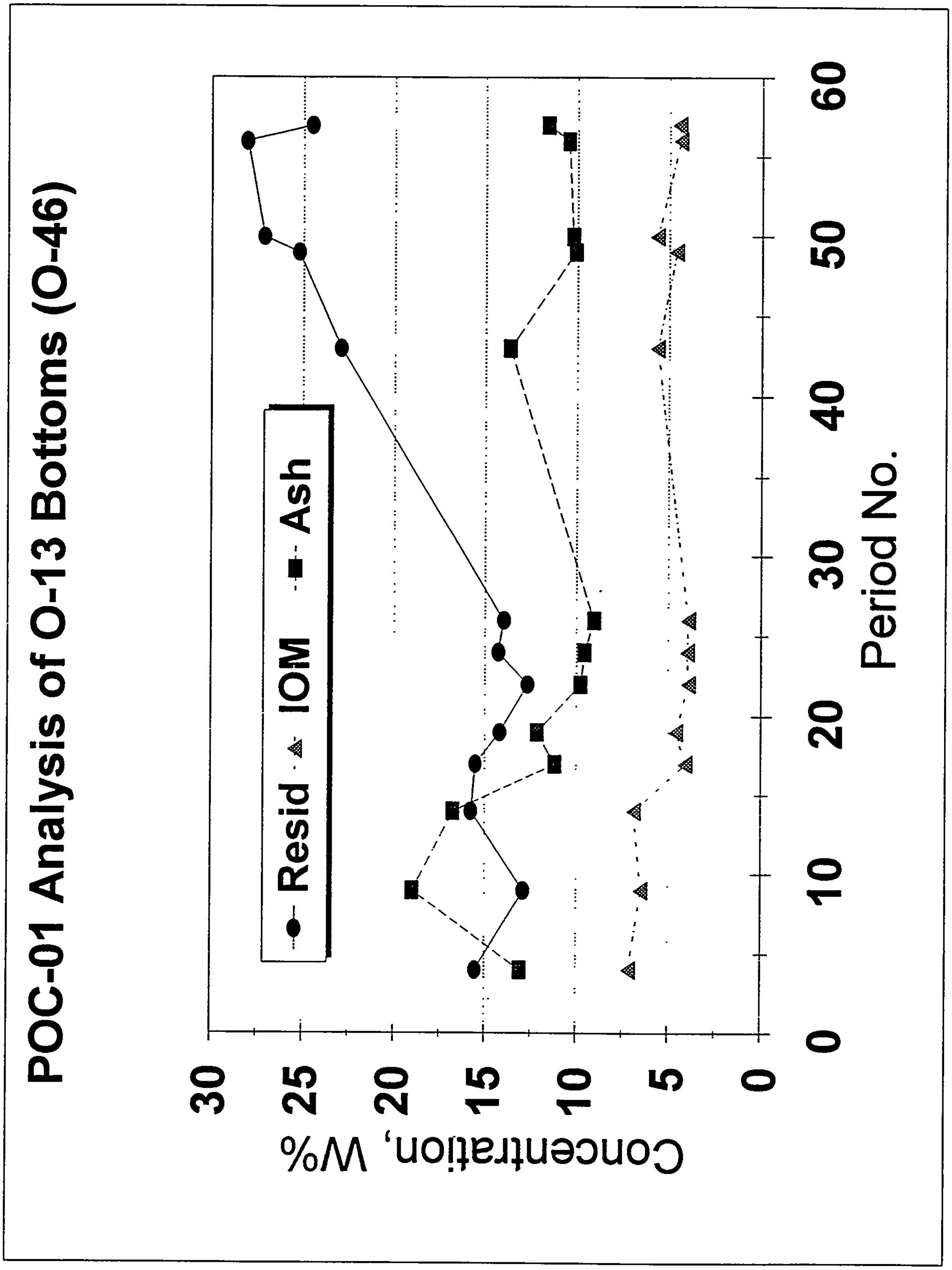
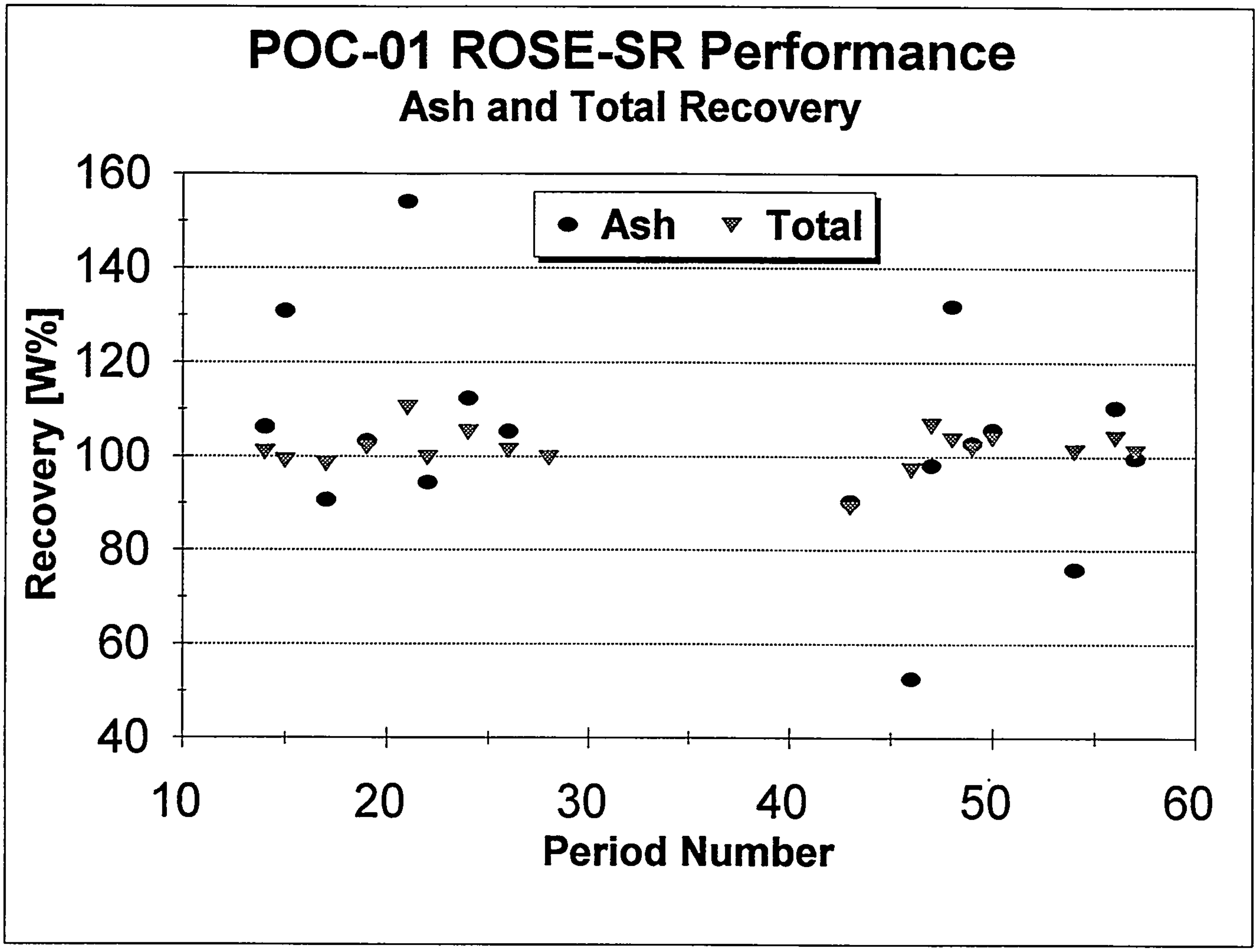
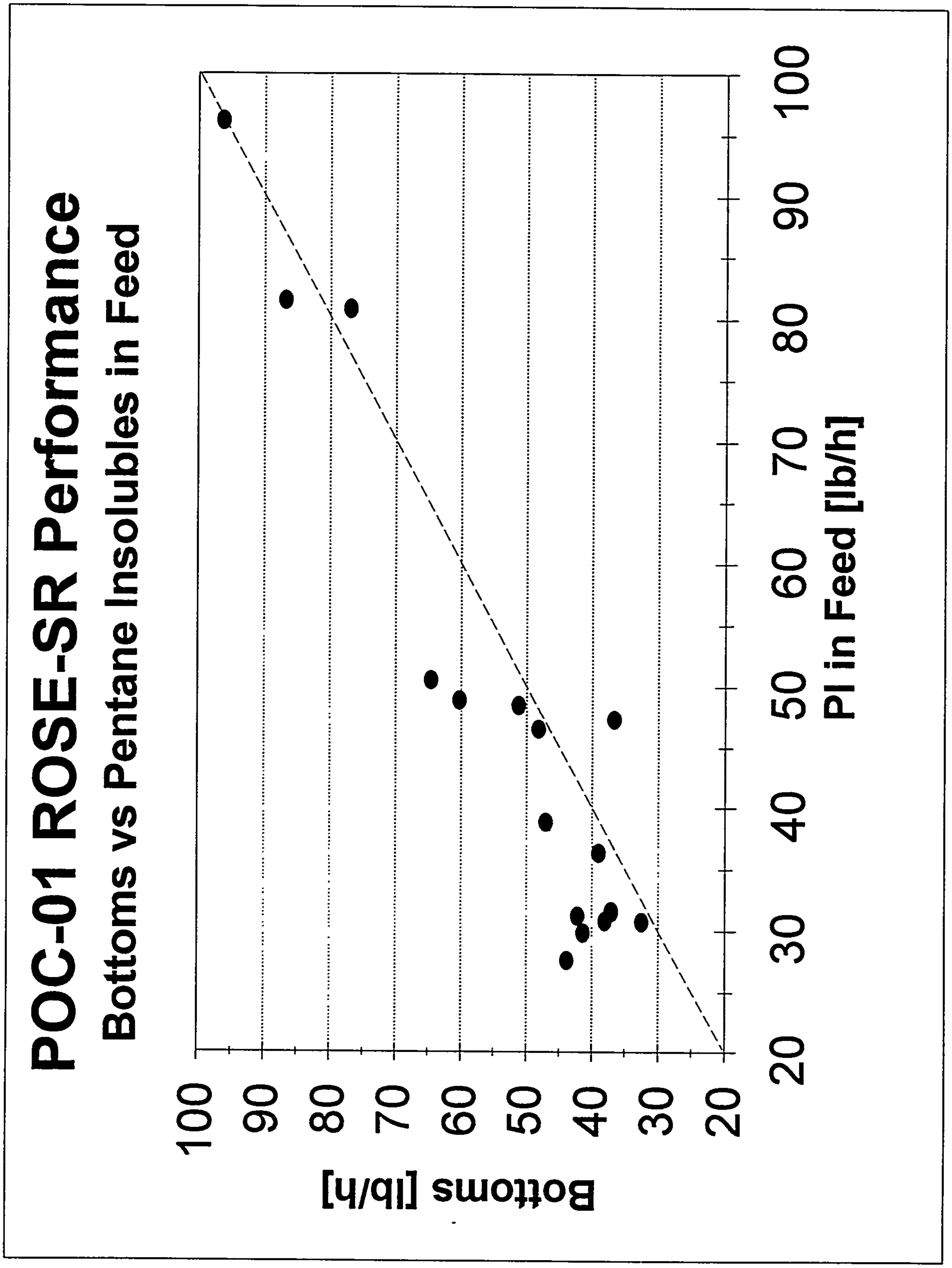
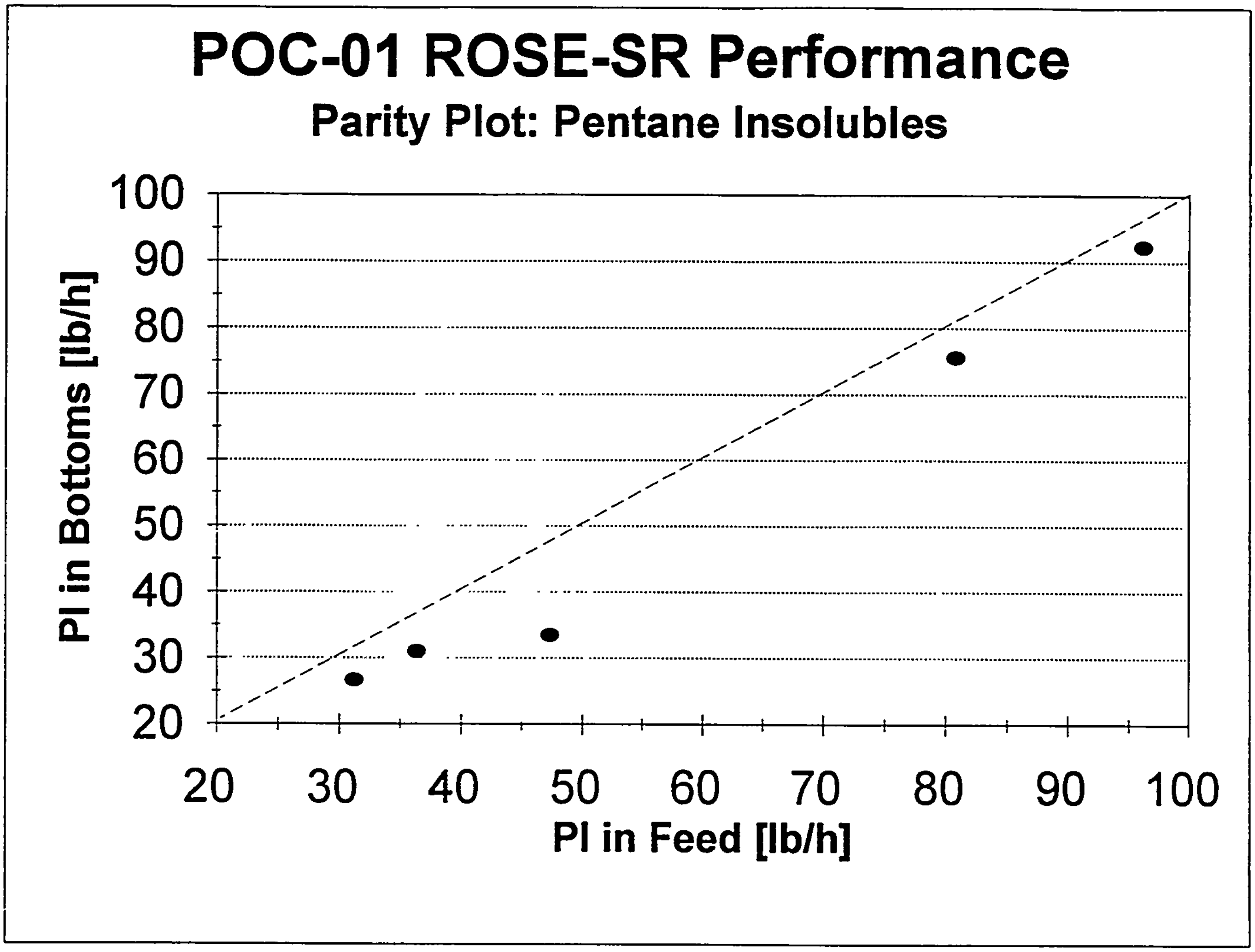

POC-01 Analysis of O-13 Bottoms (O-46)
Resid
IOM
Ash
Concentration, W%
Period No.
0
5
10
15
20
25
30
0
10
20
30
40
50
60

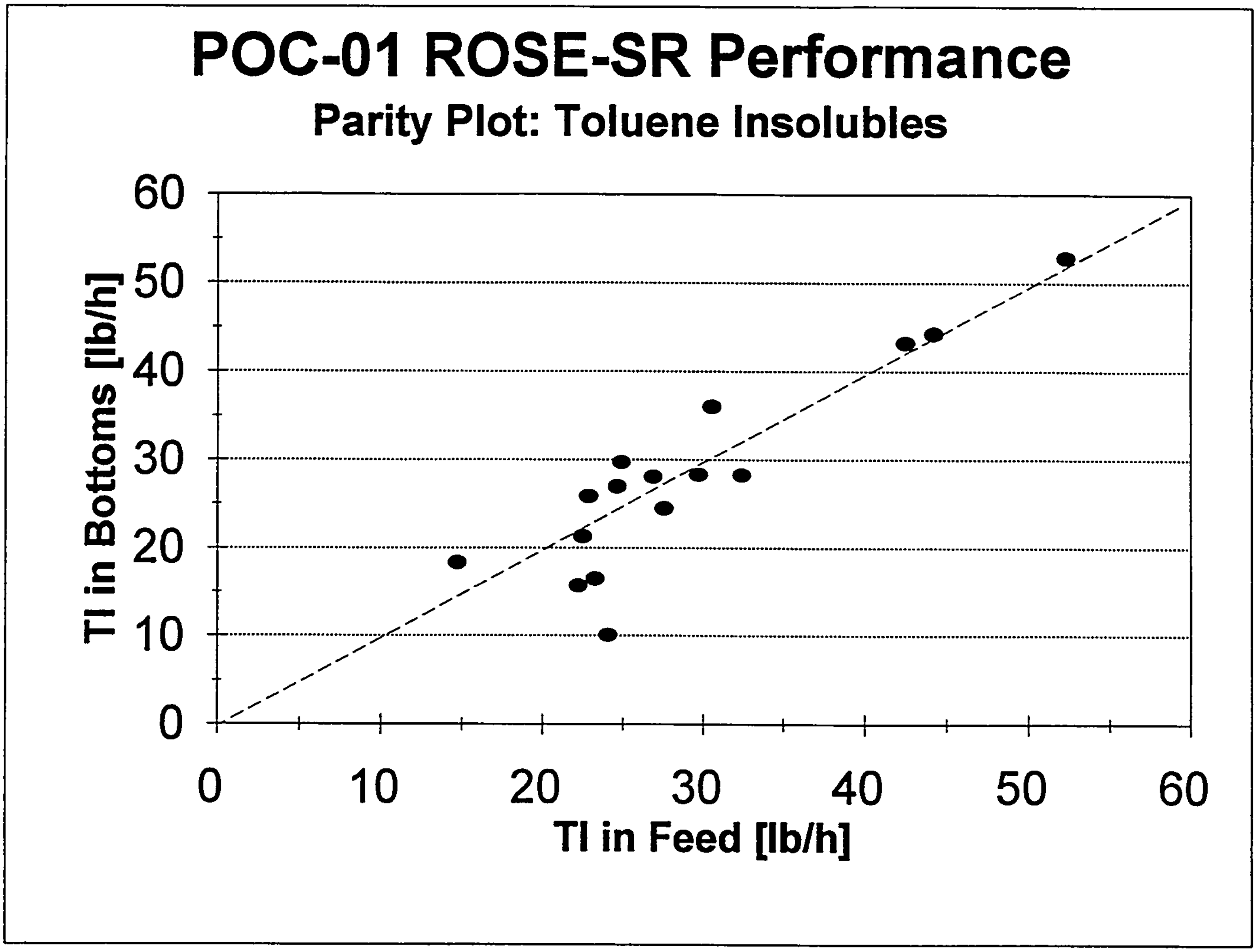
FIGURE 6.13

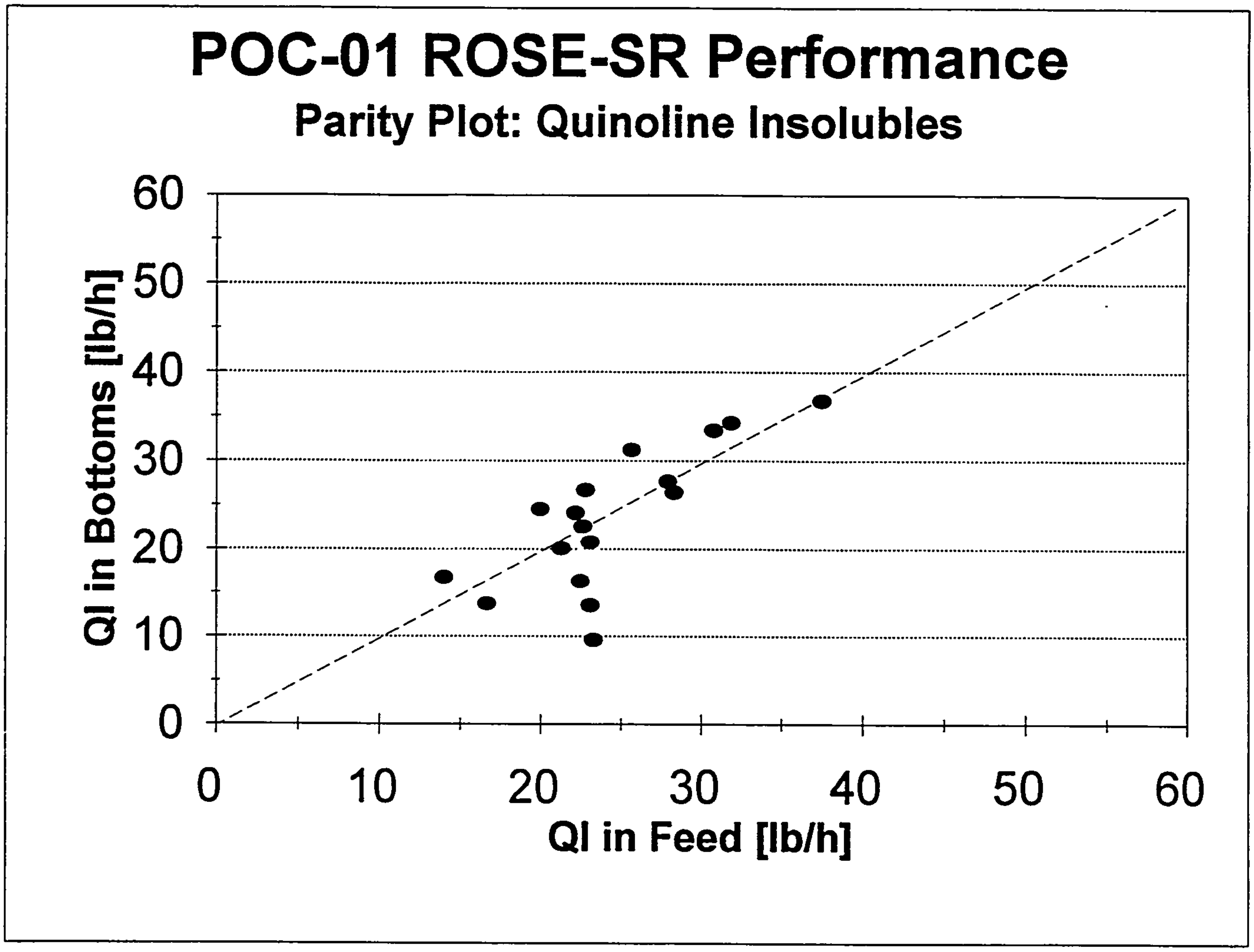

POC-01 ROSE-SR Performance
Ash and Total Recovery
Ash
Total
Recovery [W%]
160
140
120
100
80
60
40
10
20
30
40
50
60
Period Number

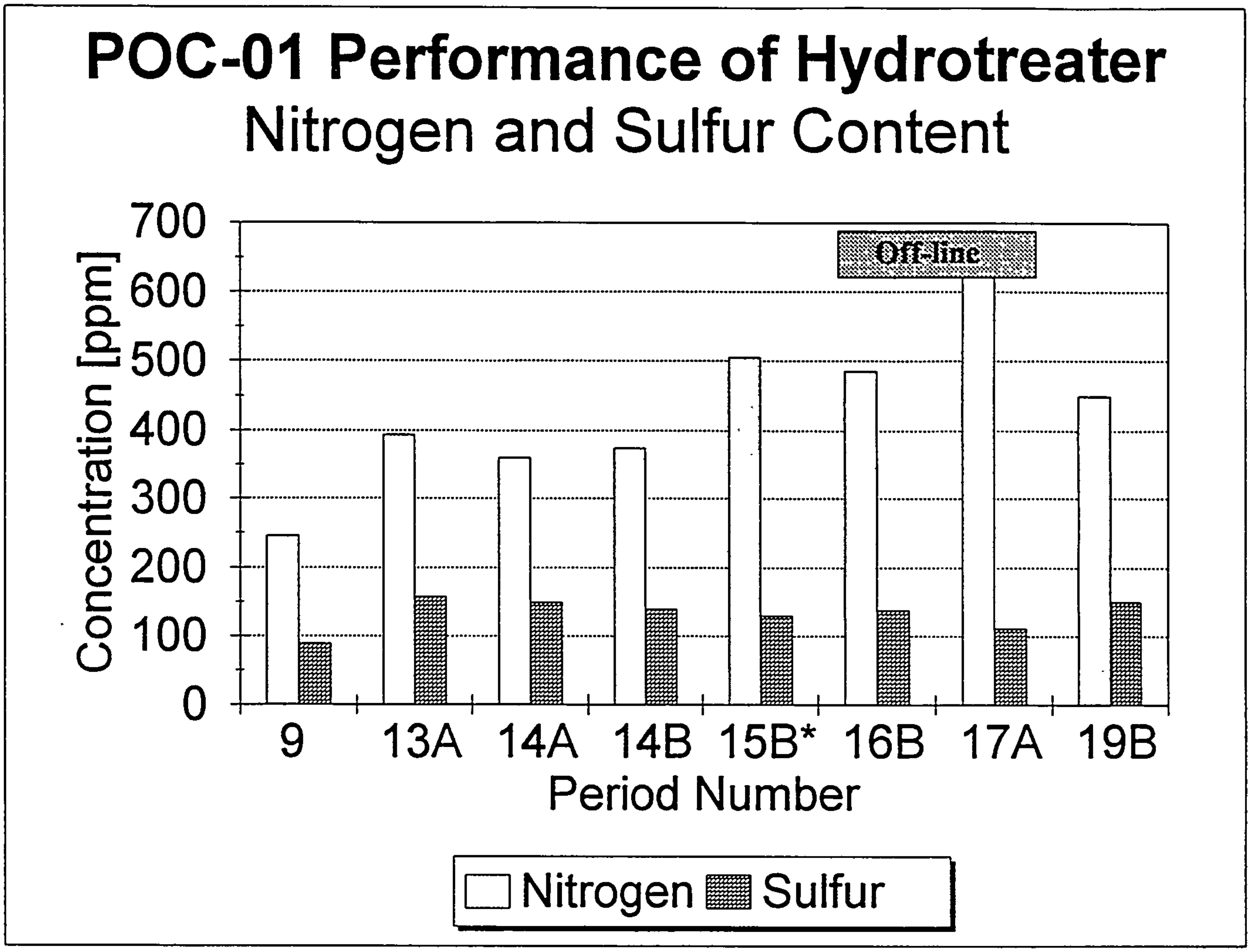

POC-01 ROSE-SR Performance
Bottoms vs Pentane Insolubles in Feed
Bottoms [lb/h]
100
90
80
70
60
50
40
30
20
PI in Feed [lb/h]



POC-01 ROSE-SR Performance
Parity Plot: Pentane Insolubles
PI in Bottoms [lb/h]
100
90
80
70
60
50
40
30
20
20
30
40
50
60
70
80
90
100
PI in Feed [lb/h]


POC-01 ROSE-SR Performance
Parity Plot: Toluene Insolubles
TI in Bottoms [lb/h]
TI in Feed [lb/h]
0
10
20
30
40
50
60


POC-01 ROSE-SR Performance
Parity Plot: Quinoline Insolubles
QI in Bottoms [lb/h]
QI in Feed [lb/h]
0
10
20
30
40
50
60



POC-01 Performance of Hydrotreater
Nitrogen and Sulfur Content
Concentration [ppm]
700
600
500
400
300
200
100
0
Off-line
9
13A
14A
14B
15B*
16B
17A
19B
Period Number
Nitrogen
Sulfur

SECTION VII

LABORATORY SUPPORT

A. COAL QUALIFICATION TESTS

A.1 Microautoclave Tests Series I

This series consisted of the standard coal qualification tests carried out on the Illinois No. 6 coal received from Burning Star Mine No. 4 (HRI-6125). Following are the test conditions and the results are shown in *Tables 7.1 and 7.2.*

Coal:	Illinois No. 6 (HRI-6125), 2.0 g
Solvent:	A number of HRI-standard solvents were used, 8.0 g
Catalyst:	Presulfided Shell-317 1/32" extrudates

A.2 Microautoclave Tests Series II

Due to unavailability of Illinois No. 6 Burning Star Mine No. 4 coal (mine-washed) from the Consolidation Coal Company, a new batch of Illinois No. 6 coal (Crown II mine (HRI-6141)), mined at a similar seam, was obtained from the Freeman United Coal Company. The same test as performed on the Burning Star Mine No. 4 coal was performed on the Crown II mine coal and these results are presented in *Tables 7.3 and 7.4.* Using HRI's standard coal qualification procedures employing microautoclave testing, comparisons were made between conversion levels of the Burning Star Mine No. 2 and No. 4 Illinois No. 6 coals (tested earlier) with the new batch of Illinois No. 6 Crown II mine coal. These results are presented in *Table 7.5.*

Based on these tests, the new batch of Illinois No. 6 (Crown II Mine) coal, obtained from the United Freeman Coal Co. (HRI-6141), seems to be almost as reactive as the Illinois No. 6 Mine 4 coal (HRI-6125) tested earlier, as indicated by the data in *Table 7.4.* Both total coal conversion (based on the THF-solubility of the products) and resid conversion for HRI-6141 coal are higher (under thermal as well as catalytic conditions) than for Illinois No. 6 Mine 2 coal (HRI-6081); while the results are similar to those for the Mine 4 coal (HRI-6125). *Table 7.5*, which compares the kinetic test data for the Mine 4 and Crown II Mine coals, shows that, although under certain conditions, Crown II Mine coal results in THF conversions about 1-2 Wt% lower than the Mine 4 coal, resid conversion levels are mostly similar or about 1-2 Wt% higher for the Crown II coal. In light of these results, selection of the Illinois No. 6 coal from the Crown II Mine (to be supplied by the United Freeman Coal Co.) as a feed coal for the POC-01 was reasonable.

B. SOLVENT QUALIFICATION TESTS

B.1 Startup/Makeup Solvent Screening Test

The standard "Equilibrium Solvent Quality" test employs solvent as the only "source" of hydrogen for coal conversion, as no hydrogen is used in the gas phase, (pure nitrogen is employed). Typical conditions used for such testing are:

3g of coal (either Illinois No. 6 or Black Thunder Mine coal)
6g of solvent to be tested
394°C (750°F) for 30 minutes at 13.8 MPa (2000 psig) of N_2

The efficacy of a tested solvent is based on the total coal conversion, based on THF solubility, obtained during the tests. *Table 7.6* presents the results of the solvent quality testing, along with some earlier solvent-quality data for comparison.

From the results in *Table 7.6* it is clear that the FCC cycle oil from Mobil (HRI-6172) was a good solvent, almost as good as HRI-5198 (HRI's standard solvent) and much better than the other petroleum-based oils tested in this series. This FCC cycle oil had an API gravity of about -8.5° and more than about 93 V% material boiling above 650°F.

B.2 Process Recycle Solvent Quality Testing

The quality of the recycle oil obtained during different periods of POC-01 was investigated as a solvent for coal conversion. This "solvent quality" signifies the H-donor ability of a solvent, which is critical for coal dissolution in the first stage reactor of the CTSL Process. Standard "equilibrium tests" were conducted to assess the solvent quality: 2:1 solvent to coal feed, 30 min reaction at 399°C (750°F) under 2000 psig of N_2. The tests were carried out in HRI's 20 cc microautoclaves using no catalysts; the two makeup solvents, namely L-803 and L-809 (hydrotreated Cat Cycle Oils), were also evaluated for their quality as solvents for coal liquefaction. Percentage coal conversion (based on THF solubility), obtained in such tests, is used to compare the qualities of the solvents.

As shown in *Table 7.7 and Figure 7.1*, the quality of the recycle oils derived from the process during POC-01 (O-43 filtered material) was, on an average, inferior to that of the two makeup solvents. During the run-periods where catalyst was being periodically replaced, the solvent quality remained about the same (about 65 Wt% maf coal conversion), while towards the end, i.e., Periods 56 and 57, when catalyst replacement was not effected due to operational problems, the recycle oil obtained was a better solvent (5-7 Wt% higher coal conversion). This is probably due to the fact that as catalyst deactivated, the recycle oil became more aromatic in nature and also contained more resid material. (This effect has been observed from time to time during bench scale operations, where catalyst undergoes a batch deactivation.)

C. HYDROTREATING SCOUTING TESTS

In CTSL processing of coal, the reactor effluent from the coal liquefaction reactors is passed to a high pressure high, temperature separator. The separator overheads (SOH) which are rich in hydrogen and contain high amounts of aromatics, sulfur and nitrogen, would normally pass through a low pressure, low temperature separator to separate off-gas and light hydrocarbon products. Since the SOH is rich in hydrogen, it can be easily hydrotreated on-line at a reduced cost to obtain clean products. Inclusion of an on-line hydrotreater in the CTSL processing scheme has, therefore, been considered to improve the product quality and the economics of the process.

C.1 Objectives

A bench scale hydrotreating test program was conducted in support of the Proof-of-Concept (POC)-Direct Coal Liquefaction Program to test the hydrotreatibility of coal derived liquids, i.e. SOH and SOH+VSOH, in a fixed-bed trickle-bed reactor. The key objectives of the bench program were;

- To obtain parameters needed for the internal design of an in-line hydrotreater.
- To obtain information on the hydrotreating process performance.
- To recommend operating conditions for the POC-1 in-line hydrotreater.

Experiments were designed to obtain the following information:

- Performance of the Criterion C-411 catalyst in hydrotreating coal derived liquids. This includes the activity/stability of the catalyst, which is needed to determine the catalyst life.
- Kinetic parameters for HDS, HDN and cracking (if any) reactions.
- Product yields and hydrogen consumption at various LHSVs.
- Effect of H_2O on catalyst performance. Since coals contain a high percentage of oxygen, liquefied coal liquids contain a high percentage of water as a result of hydrodeoxygenation reactions. The effect of water on catalyst performance may be detrimental.
- Effect of VSOH material on process performance.
- Effect of coal liquids, derived from Wyoming and Illinois coals on process performance.

C.2 Experimental

The hydrotreating experiments were conducted in a bench scale unit (Unit 246) equipped with a 2.5 cm (1 in) I.D. reactor. The reactor was packed with 50 ml (3 cu in) of Criterion C-411 catalyst. The catalyst bed was diluted with 25 ml (1.5 cu in) of alundum, an inert material, in order to have good axial dispersion of liquid and good solid-liquid contacting ("wetting"). The top and bottom sections of the catalyst bed were also packed with larger diameter (0.8 mm) (0.03 in) diluent followed by a smaller particle size (0.5 and 0.2 mm (0.02 and .008 in)) diluent in order to provide good liquid dispersion. A schematic description of the reactor is shown in *Figure 7.2.*

Feedstocks were coal derived liquids, derived from Wyoming and Illinois coal obtained form previous coal liquefaction experiments. The coal derived liquid of Wyoming origin was used as a base feedstock. The reactivities of coal liquids of Illinois origin and of Wyoming origin containing 20 V% VSOH were also tested. The properties of these feedstocks are given in *Table 7.8.* Adequate amounts of tertiary butyl-amine (TBA), di-methyl-di-sulfide (DMDS) and water were added to the feedstock to produce sufficient quantities of NH_3 and H_2S to simulate coal liquefaction reactor effluent.

The Criterion C-411 catalyst was chosen for this duty because this catalyst was designed for light feedstocks and has high hydrodenitrogenation activity. The catalyst was treated using the standard HRI pretreatment procedure (H-119) and sulfided in-situ.

The experimental program and operating conditions are summarized in *Table 7.9*. The specific objectives of the operating condition are as follows:

Condition	Objectives
1-4	Catalyst Stabilization
1	Start-up condition
2	Temperature scouting to determine Arrhenius parameters
3	Temperature scouting to determine Arrhenius parameters
4	Temperature scouting to determine Arrhenius parameters
5	Base line at LHSV of 1 h^{-1}
6	Space velocity scouting to determine product yields and hydrogen consumption after each catalyst bed
7	Space velocity scouting to determine product yields and hydrogen consumption after each catalyst bed
8	Space velocity scouting to determine product yields and hydrogen consumption after each catalyst bed
9	Base activity check
10	Temperature scouting to study aromatics hydrogenation
11	Feedstock scouting - Hydrotreating of coal liquids of Illinois origin. (Instructions on feedstock preparation are given later.)
12	Feedstock scouting. Inclusion of VSOH in the feedstock. (Instructions on feedstock preparation are given later.)
13	Base activity check.

Detailed experimental data resulting from hydrotreating experiments are presented in Appendix E.

C.3 Activity/Stability of C-411 Catalyst

The activation energy and frequency factor for the HDN reactions were calculated to be 29.3×10^3 j/mol btu/mol and 9.2×10^5 h^{-1}, respectively. *Figure 7.3* shows the required operating temperature (ROT) for 10 ppm nitrogen slip (nitrogen content of the outlet stream from the hydrotreater) for the hydrotreated coal liquids vs. catalyst age. The rate of deactivation for the HDN reactions for the initial 600 hours of operation was calculated to be 33°C/1000h (59°F/1000h). After 600 hours of operation, the rate of catalyst deactivation levels off and is calculated to be 6.1°C/1000 h (11°F/1000h). The start-of-run temperature is calculated to be 379°C (714°F). The coal liquids of Illinois origin (L-791) was found to be 11.1°C (20°F) more reactive (temperature difference required in order to maintain the same nitrogen slip) than that of Wyoming origin. Inclusion of 20 V% of coal derived VSOH in the SOH feedstream decreased the reactivity by 7.2°C (13°F). Based on an estimated end-of-run temperature of 427°C, the life of the C-411 catalyst is calculated to be at least one year.

Because of the scatter in the sulfur removal data, an activity/stability curve for HDS reactions could not be drawn. Scatter is assumed to be due to contamination by H_2S and mercaptans.

Cracking of the 249°C$^+$ (480 °F$^+$)fraction was minimal and, therefore, is not presented here.

C.4 Product Quality

Hydrotreated coal liquid products were inspected to obtain product quality data. Initially, the total liquid products were fractionated into several cuts, IBP-82°C (^{IBP}-180°F), 82-177°C (180-350°F), 177-249°C (350-480°F), 249-343°C (480-650°F), 343°C$^+$ (650°F+), using ASTM D86. *Figures 7.4 and 7.5* illustrate the sulfur and nitrogen distributions, respectively, obtained at various operating temperatures and catalyst ages. As seen from these figures, both sulfur and nitrogen content of the fractions boiling above 177°C (350°F) were decreased substantially. Note that high sulfur contents of the fractions boiling below 177°C (350°F) are probably due to contamination by H_2S and mercaptans. Aromaticity of the hydrotreated products was also monitored during the program. *Figure 7.6* depicts the aromatic contents of the products versus operating temperature. It is seen that the aromatic content of the products obtained at early catalyst age decreases with increasing operating temperature (360°C to 379°C (680-714 °F)). However, a further increase in temperature did not affect the hydrogenation reactions and resulted in high aromatics content in the products, suggesting that hydrogenation reactions are thermodynamically limited. Aromaticity of hydrotreated products obtained at 379°C and at catalyst ages of 72 h and 624 h are also compared in *Figure 7.6.* The increase in the aromatic content of the products clearly shows that the selectivity of the catalyst changes as the catalyst deactivates.

For the hydrotreated products from periods 40/41, 47/48 and 52/53, TPB (true boiling point) distillations using ASTM D-2892 were performed. It should be noted that the effect of feedstocks was studied during these periods: SOH of Wyoming origin in periods 40/41, SOH of Illinois origin in periods 47/48, and SOH and VSOH of Wyoming origin in periods 52/53. The TBP distillations were performed to generate sharp boiling point fractions which were subsequently analyzed to determine product qualities. Two fractions, consisting of IBP-177°C (IBP-350°F) and 177-343°C (350-650°F) were distilled. Detailed analyses performed on these blended fractions are provided in *Table 7.10.* The cetane number for the coal liquids of Illinois origin was 2 points higher than that of Wyoming origin. Inclusion of VSOH in the SOH fraction did not affect the cetane number of the products. The smoke points were found to be in the range of 14-16 mm.

Research and motor octane numbers (RON and MON) for the IBP-177°C (350°F) fractions were also determined. The RON numbers were 60.8, 68.1, and 59.5 for feedstocks L-790, L-791 and L-792, respectively.

It can be concluded that coal liquids can be hydrotreated to obtain sulfur and nitrogen free products. However, the quality of the hydrotreated products in terms of cetane number, smoke point, and octane number, is poor.

Table 7.1 Standard Coal Qualification Testing				
Reaction Conditions: 427°F for 30 minutes at 13.8 MPa H_2				
	THF Conversion, Wt% Coal			975°F+ Resid Conversion, Wt%
Standard Solvent Used	B.S. Mine No. 2, HRI-5174	B.S. Mine No. 2, HRI-6081	B.S. Mine No. 4, HRI-6125	B.S. Mine No. 4, HRI-6125
HRI-5198	91.0	91.7	96.2, 96.3*	59.4, 63.0*
HRI-6002			96.2, 96.6*	63.3, 60.1*
RUN 227-78-Period 05 PFL			98.1, 98.3*	46.4, 45.9*

* These were the values obtained in duplicate experiments.

Table 7.2 Kinetic Tests on Burning Star Mine No. 4 Illinois Seam No. 6 (HRI-6125) Coal			
Temperature, °C	Time, min	THF Coal Conversion, Wt%	975°F+ Resid Conversion
399	30	91.5, 91.6	47.0, 50.6
427	15	95.3, 94.3	54.1, 56.8
427	60	97.0, 92.4	69.4, 60.7
440	30	93.7, 96.5	66.9, 72.7

Table 7.3. Standard Coal Qualification Testing for the Crown II Mine Illinois No. 6 Coal			
Reaction Conditions: 427°C for 30 minutes at 13.8 MPa H_2			
Solvent	Catalyst Used	THF Coal Conversion, Wt%	975°F Resid Conversion, Wt%
HRI-5198	Yes	95.2	66.3
HRI-5198	No	89.6	36.1
HRI-6002	Yes	87.9	55.5
HRI-6002	No	83.6	28.6
Run 227-78-Period 05 PFL	Yes	91.0	39.4
Run 227-78-Period 05 PFL	No	88.0	22.9

Table 7.4 Kinetic Tests on Crown II Mine Illinois Seam No. 6 (HRI-6141) Coal*			
Temperature, °C	Time, min	THF Coal Conversion, Wt%	975°F$^+$ Resid Conversion
399	30	90.9 (91.5)	51.9 (47.0)
427	15	90.6 (94.3)	58.6 (56.8)
427	60	94.1 (94.7)	69.2 (65.1)
440	30	89.6 (93.7)	65.0 (66.9)

* The numbers in the parentheses are the values of conversions for HRI-6125 Burning Star Mine No. 4 Illinois No. 6 coal.

Table 7.5 Comparison of Coal Reactivity Results for Three Illinois No. 6 Coals				
Reaction Conditions: 2.0 g Coal, 8.0 g Solvent (HRI-5198), 2.0 g Presulfided Shell-317 Catalyst, 13 MPa H_2, 427°C and 30 min Reaction Time				
Illinois No. 6 Coal From:	HRI Number	Catalyst Used	THF Coal Conversion, Wt%	975°F$^+$ Resid Conversion, Wt%
Burning Star Mine No. 2	6081	Yes	91.7	57.5
Burning Star Mine No. 4	6125	Yes	96.2	61.2
Crown II Mine	6141	Yes	95.2	66.3
Burning Star Mine No. 2	6081	No	86.5	34.1
Crown II Mine	6141	No	89.6	36.1

Table 7.6 Results of the Standard Equilibrium Solvent Quality Testing		
Solvent Tested	THF Coal Conversion for Illinois No. 6, Wt%	THF Coal Conversion for Black Thunder Mine Coal, Wt%
Topped L-769	49.9	52.2
HRI-5669	29.0	31.1
HRI-5737	32.4	36.8
HRI-5667	29.3	N/A
HRI-6172	52.0	55.0
HRI-5198	55.5	56.2
L-799	56.77	45.54

Table 7.7
POC-01 Solvent Quality Tests

Coal: Illinois No. 6 Crown II Mine Coal
Temperature: 399°C (750°F)
Pressure: 13.8 MPa (2000 psig)
No. Catalyst: Solvent to Coal 2:1

				% maf Coal Conversion
Startup/Makeup Solvent:				
L-803 (First batch)				69.4
L-809 (Second batch)				77.0
Filtered Recycle (O-43 oil) Solvent:				
Condition	**Day in Condition**	**Period**	**% Makeup Used**	
L/O	4	4	38.3	60.2
L/O	4	9	36.3	61.5
L/O	2	12	21.4	61.7
1	5	17	0.0	64.0
1	7	19	3.7	62.0
2	5	24	2.7	63.0
2	7	26	0.0	65.1
L/O	2	40	0.0	60.0
3B	2	42	24.2	60.0
3B	3	43	6.6	57.0
L/O	2	46	1.3	63.0
4A/B	4	50	0.0	63.5
4C	3	56	3.5	70.4
4C	4	57	1.6	69.2

Table 7.8
PROPERTIES OF UNHYDROTREATED COAL LIQUIDS

Feedstocks			L-790		L-791		L-792	
Feedstock Origin			Wyoming		Illinois		Wyoming	
Feedstock Type	*Units*	*Method*	SOH		SOH		SOH + VSOH	
Gravity	*°API*	*D287*	33.2		39.6		28.4	
C	*W%*	*H104*	86.28		86.2		86.85	
H	*W%*	*H104*	12.49		13.16		12.13	
S	*ppm*	*D4239*	937		297		569	
N	*ppm*	*D3228*	2547		161		1001	
[1]PONA (IBP-177 ° C), [2]PNA (177-343 °C)		*1, 2*	IBP - 177 °C	177 - FBP °C	IBP - 177 °C	177 - FBP °C	IBP - 177 °C	177 - FBP °C
Paraffins	*V%*		28.8	9.9	24.3	12.2	25.8	11.3
Olefins	*V%*		0.3	----	0.3	-----	0.3	-----
Naphthenes	*V%*		64.2	43.6	67.0	48.2	64.1	38.3
Aromatics	*V%*		6.7	46.5	8.4	39.6	9.8	50.34
Cetane Index		*ASTM Corr.*	36.4		38		37.3	
Distillation		*D86*						
IBP / 5	*°C*		74 / 108		62 / 92		96 / 136	
10 / 20	*°C*		124 / 158		103 / 118		161 / 201	
30 / 40	*°C*		194 / 221		136 / 161		232 / 261	
50 / 60	*°C*		246 / 262		191 / 217		281 / 301	
70 / 80	*°C*		279 / 296		252 / 279		324 / 347	
90 / 95	*°C*		320 / 434		311 / 337		369 / 386	
FBP	*°C*		378		370		386	

1 PONA by Mass Spec. & FIA (ASTM D-2789)
2 PNA by Mass Spec. (ASTM D-2425)

Table 7.9
RUN PLAN FOR 246-238

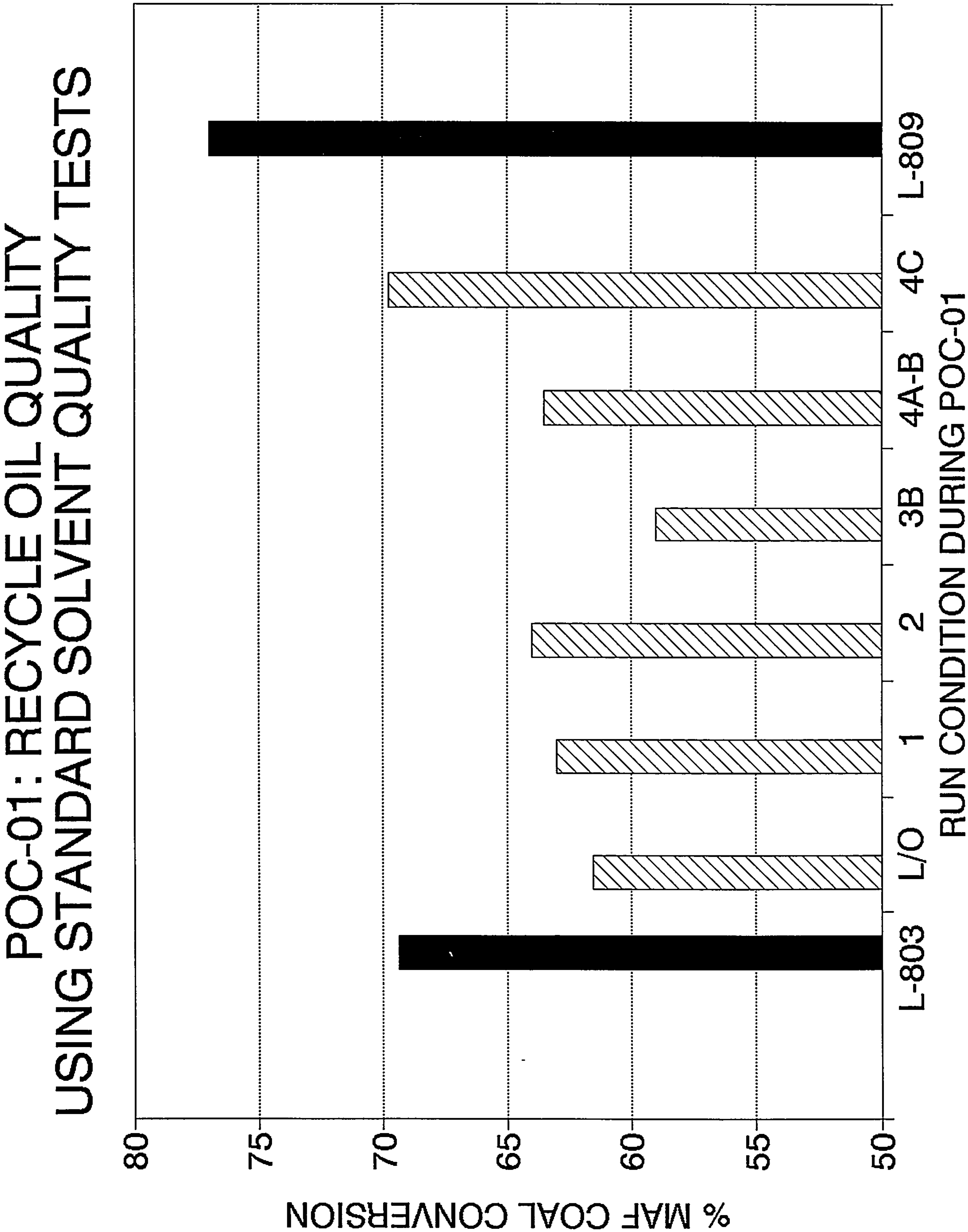
Condition	1	2	3	4	5	6	7	8	9	10	11	12	13
Period	0-12	13-15	16-18	19-21	22-26	27-30	31-34	35-38	39-41	42-44	45-49	50-54	55-57
Objective	ST	ST	ST	ST									
	SU	T	T	T	Base	LHSV	LHSV	LHSV	Base-Check	Temp.	Feed-stock	Feed-stock	Base-Check
LHSV, h^{-1}	1	1	1	1	1	2	3	4	1	1	1	1	1
Temperature, °C	379	393	360	379	379*	379*	379*	379*	379	385	379*	379*	385
Gas/Oil Ratio,m^3/bbl oil	156	156	156	156	156	156	156	156	156	156	156	156	156
Inlet H_2 Pressure, MPa	12.4	12.4	12.4	12.4	12.4	12.4	12.4	12.4	12.4	12.4	12.4	12.4	12.4
Oil Feed Rate, g/h	50	50	50	50	50	100	150	200	50	50	50	50	50
Water Rate, g/h	7	7	7	7	7	14	21	28	7	7	7	7	7
H_2 Feed, m^3/h (10^{-2})	5	5	5	5	5	10	15	20	5	5	5	5	5

ST Catalyst stabilization
T Temperature scout
LHSV Space velocity scout
* or temperature required to obtain targeted conversion

Table 7.10
PROPERTIES OF HYDROTREATED COAL LIQUIDS (RUN 246-350)

Periods	Units	Method	40 / 41	40 / 41	47 / 48	47 / 48	52 / 53	52 / 53
Catalyst Age	h		972	972	1140	1140	1260	1260
Feedstock #			L-790	L-790	L-791	L-791	L-792	L-792
Feedstock Origin			Wyoming	Wyoming	Illinois	Illinois	Wyoming	Wyoming
Feedstock Type			SOH	SOH	SOH	SOH	SOH + VSOH	SOH + VSOH
Fractions			IBP - 177 ° C	177 - 343 ° C	IBP - 177 ° C	177 - 343 ° C	IBP - 177 ° C	177 - 343 ° C
Wt Fraction			0.2737	0.6803	0.4284	0.5332	0.2125	0.6569
Gravity	°API	D287	51.8	28.8	51.4	30.8	51.2	28.2
C	W%	H104	85.66	87.48	85.78	87.21	85.72	87.57
H	W%	H104	14.34	12.52	14.22	12.79	14.28	12.43
S	ppm	D4239	1.7	13.5	3.7	13.9	3	38.3
N	ppm	D3228	<1.0	9.5	1.0	<1.0	<1.0	6.0
[1]PONA (IBP-177 ° C), [2]PNA (177-343 ° C)		1, 2						
Paraffins	V%		26.4	11.8	22.8	13.7	24.4	13.5
Olefins	V%		0.7	-----	0.5	-----	0.8	-----
Napthenes	V%		67.0	53.0	69.3	55.7	67.5	49.5
Aromatics	V%		5.9	35.2	7.4	30.6	7.3	37.0
Cetane Number		D613		37.5		39.0		37.2
Cetane Index		ASTM Corr.		37.7		39.5		35.9
RON		D2679	58.2		62.3		58.2	
MON		D2700	60.8		68.1		59.5	
Aniline Point	°C	D1012		47.2		48.9		45.6
Smoke Point	mm	D1322		14.9		15.9		13.9

1 PONA by Mass Spec. & FIA (ASTM D-2789)
2 PNA by Mass Spec. (ASTM D-2425)
D ASTM Method
H HRI Method


POC-01 : RECYCLE OIL QUALITY
USING STANDARD SOLVENT QUALITY TESTS
% MAF COAL CONVERSION
80
75
70
65
60
55
50
L-803
L/O
1
2
3B
4A-B
4C
L-809
RUN CONDITION DURING POC-01

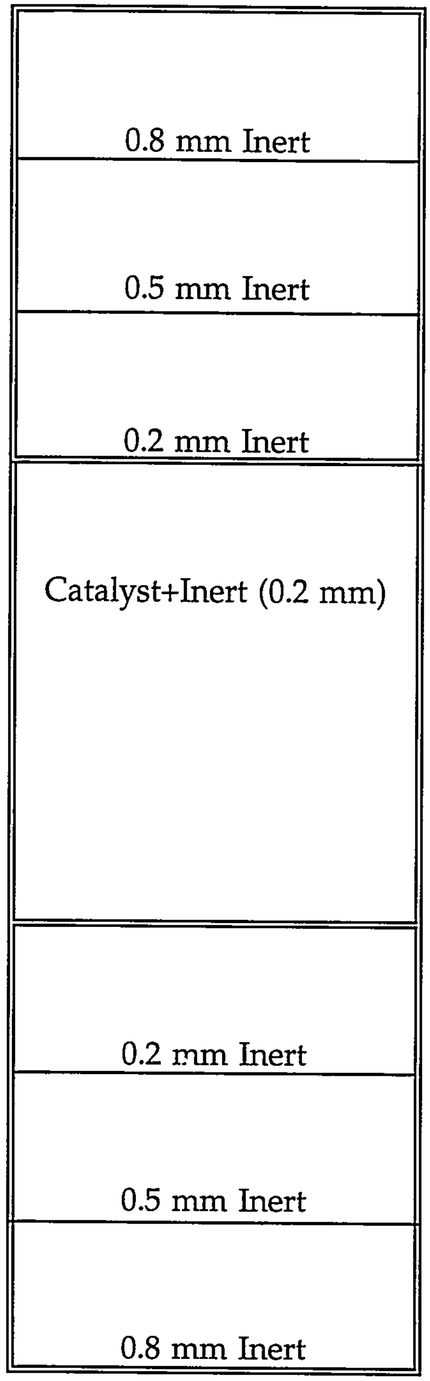
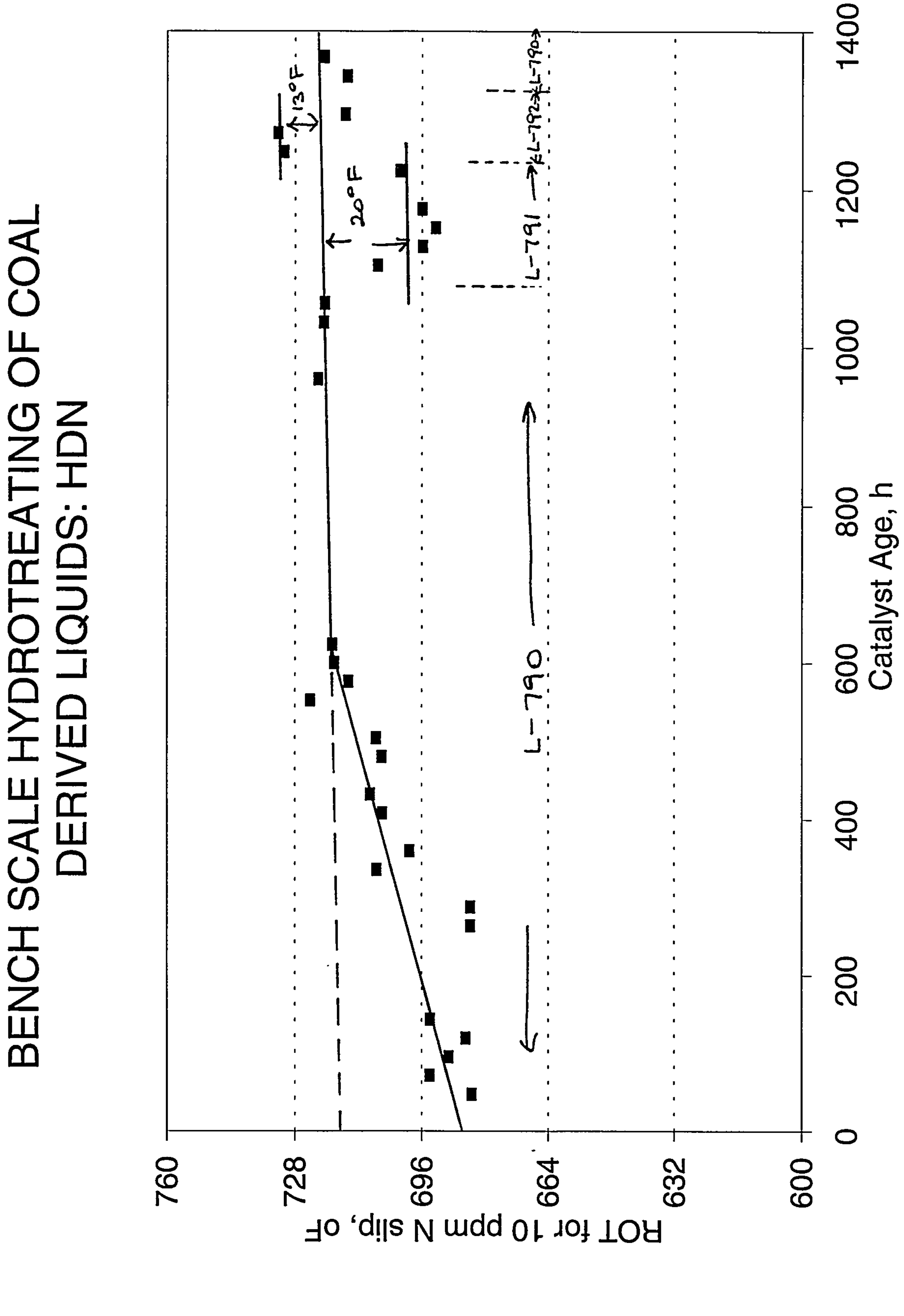
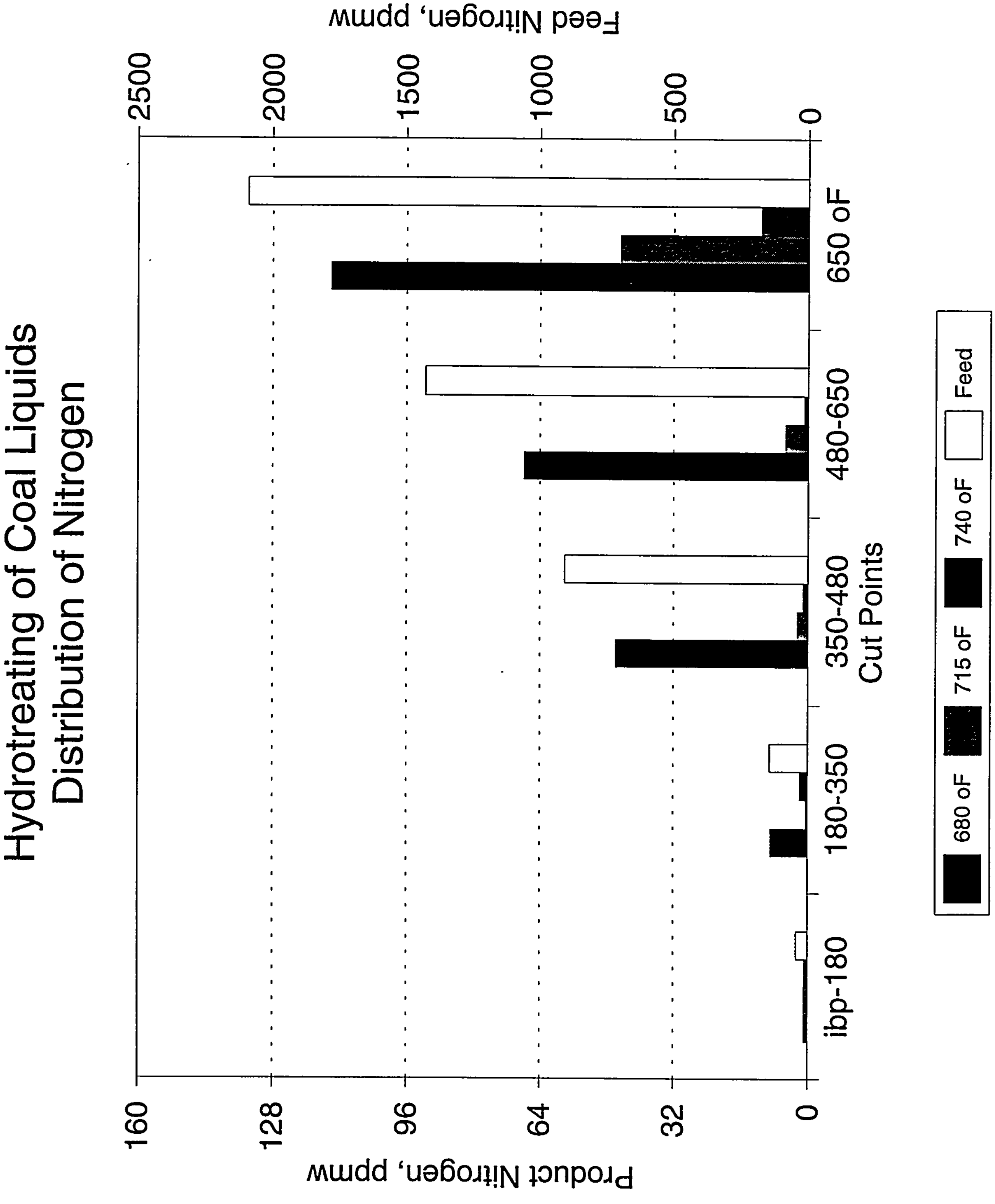


Figure 7.2 - Reactor Packing Diagram

Figure 7.3

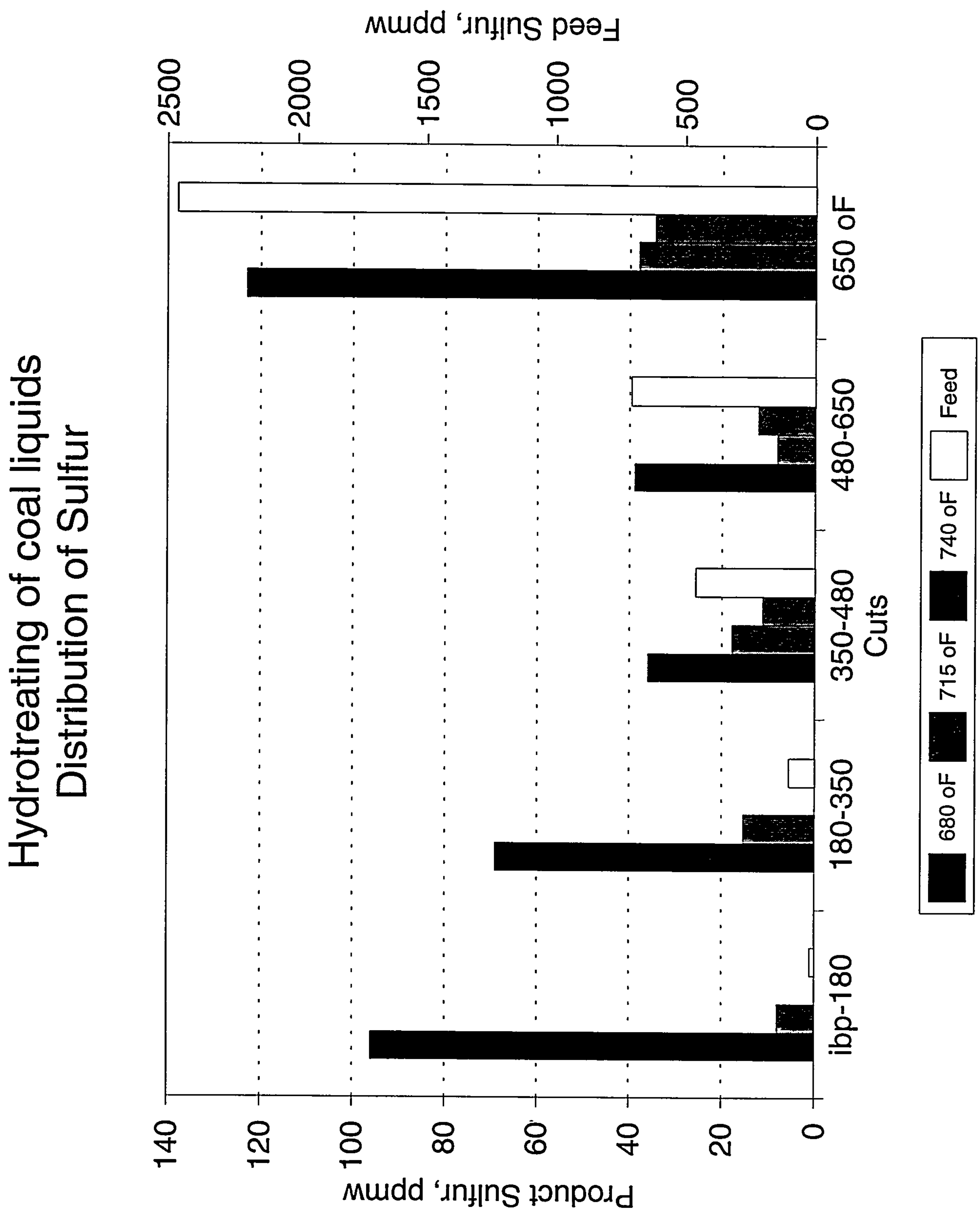


BENCH SCALE HYDROTREATING OF COAL DERIVED LIQUIDS: HDN
ROT for 10 ppm N slip, oF
760
728
696
664
632
600
Catalyst Age, h
0
200
400
600
800
1000
1200
1400
L-790
L-791
L-792
L-790
20°F
13°F



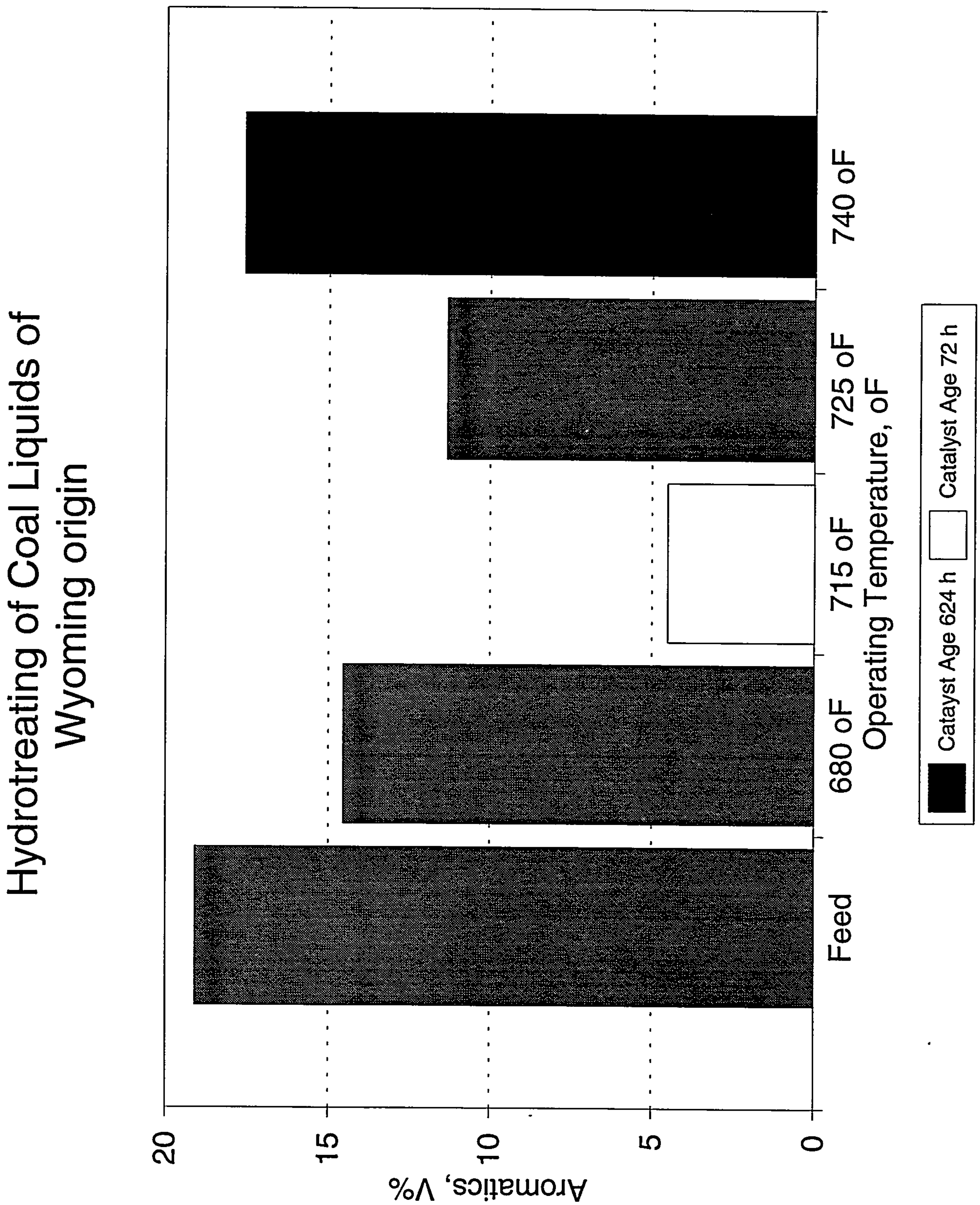
Hydrotreating of Coal Liquids
Distribution of Nitrogen
Product Nitrogen, ppmw
160
128
96
64
32
0
Feed Nitrogen, ppmw
2500
2000
1500
1000
500
0
ibp-180
180-350
350-480
480-650
650 oF
Cut Points
680 oF
715 oF
740 oF
Feed

Figure 7.5



Hydrotreating of coal liquids
Distribution of Sulfur
Product Sulfur, ppmw
Feed Sulfur, ppmw
0
20
40
60
80
100
120
140
0
500
1000
1500
2000
2500
ibp-180
180-350
350-480
480-650
650 oF
Cuts
680 oF
715 oF
740 oF
Feed

Figure 7.6


Hydrotreating of Coal Liquids of
Wyoming origin
Aromatics, V%
20
15
10
5
0
Feed
680 oF
715 oF
725 oF
740 oF
Operating Temperature, oF
Cataayst Age 624 h
Catalyst Age 72 h

SECTION VIII

TECHNICAL ASSESSMENT

A. INTRODUCTION

HRI was awarded a contract in 1992 to operate a Proof-of-Concept (POC) program to demonstrate the direct coal liquefaction (CTSL) process for the U.S. Department of Energy. The objective of the program is to develop coal liquefaction technology to produce premium coal-derived liquid fuels that are economically competitive with petroleum based fuels. As part of the POC program, HRI is performing technical assessments to provide technical, engineering and economic guidance for the POC Process Development Unit (PDU) operations.

Success of the POC program is directly related to the economics of the CTSL technology. Numerous design and economic studies have been completed in the past. In September 1992, Bechtel developed a design for the direct liquefaction of Illinois No. 6 coal. The design was based on the process performance demonstrated at the Wilsonville coal liquefaction facility. This study established the baseline for direct coal liquefaction plant design and economics. In 1988, HRI completed a design and economics study for the CTSL process using Illinois No.6 coal. This study was based upon the process performance demonstrated in bench-scale Run No. 227-47 (I-27). The study was done to assess the economic impact of improvements to the CTSL technology demonstrated in bench-scale testing at HRI.

HRI updated the 1988 design and economic study in December 1993. The purpose of the 1993 study was to develop a procedure for the evaluation of economics for the POC program and to make certain that this tool provides results that are consistent with the Bechtel baseline design study. This report provides the design and economic results based upon the first POC PDU operation (POC-01) using the procedure and economic basis developed in 1993.

B. OBJECTIVES AND SCOPE OF WORK

The objective of this study is to provide the design and economics results for the CTSL process based upon the performance from Run POC-01 (260-04). The results of HRI's December 1993 economics study (Base Case) have been included as a comparison. The scope of work is as follows:

- Develop a design basis for two operating conditions from POC-01

- Prepare a conceptual design of a grassroots CTSL plant to process Illinois No. 6 coal based upon the design basis for the two POC operating conditions

- Calculate the capital and operating costs for the CTSL plant

- Calculate the CTSL product costs and overall assessment of economics, including a comparison with the base case

C. BASIS OF DESIGN AND ASSUMPTIONS

The CTSL conceptual commercial plant design is based on the performance demonstrated in the Process Development Unit (PDU) 260, run number 6. This run is also referred to as POC-01. A simplified flow diagram of the PDU is shown in *Figure 8.1.* A detailed process description of the CTSL PDU configuration is provided in Section IV.

Coal feed during POC-01 was washed, Illinois No. 6 coal from the Crown II mine. The design coal feed is equivalent to the coal feed for POC-01. The analysis of the coal is shown in *Table 8.1.* The coal feed analysis from the base case study is also provided in the table as a reference. The coal for POC-01 was obtained from the Crown II mine, whereas the coal for the base case was obtained from the Burning Star mine. The ash content of the POC-01 coal is slightly less than the base case coal. However, the sulfur content of the POC-01 feed coal is higher. The ash and sulfur contents of the base case coal are 11.50 and 3.20 Wt% versus 10.40 and 4.17 Wt% for the POC-01 coal, respectively.

For this study, a design basis was developed for two different operating conditions (Conditions 2 and 3B) from POC-01. The actual and design operating conditions, process yields, and process performance are shown in *Tables 8.2 and 8.3* for Periods 26 (Condition 2) and 43 (Condition 3B), respectively. Additionally, the base case data are presented in the tables.

Operating conditions during Periods 26 and 43 were only slightly different from the base case. Space velocities during Periods 26 and 43 were 19.4 and 24.2 $lb/hr/ft^3$ of reactor. For the base case, the space velocity was 20.0 $lb/hr/ft^3$. The solvent-to-coal ratio was 1.15 for the base case. During Periods 26 and 43, it was 1.26 and 1.31, respectively. In addition, the residual oil content of the recycle solvent was more than six times greater in the base case. The first stage temperature was roughly the same for both periods and the base case at about 765 to 770°F. During

Periods 26 and 43 the second stage temperature was maintained at roughly 810°F. The second stage temperature for the base case is 823°F. The catalyst replacement rates for Periods 26 and 43 are based upon the actual replacement rates used during POC-01. Total catalyst replacement rates for Period 26, 43 and the base case are 4.8, 4.5 and 2.7 lb/ton, respectively.

The process yields shown for Periods 26 and 43 are normalized based on ASTM distillation data (D-86 and D-1160). For the purposes of this design and economics study, the ASTM distillation yields were converted to True Boiling Point (TBP) yields for each period. In addition, process yields for each case have been elementally balanced. The actual POC-01 and design basis yields of residuum (975°F+) and unconverted coal are the same for each period.

Coal conversion was roughly 95.0 Wt% during Periods 26 and 43. Resid conversion was 86.4 and 83.8 Wt% for the same periods. Coal and resid conversion for the base case are 95.2 and 92.0 Wt%, respectively. The C4-975°F yield for the base case was slightly higher than Periods 26 and 43. The C4-975°F yields were 64.8 and 62.7 Wt% for Periods 26 and 43, and 66.9 Wt% for the base case. However, the hydrogen efficiency was about the same for all three cases.

D. PLANT CONFIGURATION AND OVERALL DESIGN

The CTSL commercial plant design is based on prior plant design studies performed by HRI. For these current economic studies, this design information has been updated to the greatest extent possible using information from the Baseline Design performed by Bechtel. There are, however, some exceptions which need to be noted. The design configuration used in the current study produces finished quality products (gasoline and diesel) instead of synthetic crude oil in the Baseline Design. In addition, the current study is based on purchasing washed coal instead of run-of-mine coal. Finally, steam reforming is used for the manufacture of hydrogen, instead of partial oxidation. It should also be noted that the Baseline Design had evaluated most of these exceptions in sensitivity studies, and found that these options provided improved economics.

A block flow diagram of the CTSL plant's major processing areas is shown in *Figure 8.2*. The complete grassroots commercial plant design includes the following plant facilities:

AREA	DESCRIPTION
100	Coal Preparation
200	Liquefaction
300	Hydrogen Manufacturing
400	Oxygen Plant
500	Product Treating
600	Product Upgrading
700	Utilities
800	Tankage
900	General Offsites

Coal is received and prepared in the coal preparation area (Area 100). The prepared coal is sent to the liquefaction section (Area 200). The liquefaction section bottoms are sent to a deashing unit (ROSE-SR^{SM}), which is included to provide a solids-free recycle slurry oil. The bottoms from the deashing unit are fed to the partial oxidation (POX) unit (Area 300). If additional hydrogen is required, it is produced via steam reforming of natural gas in Area 300. The hydrogen produced in Area 300 is used in Area 200 and the product upgrading section (Area 600). The oxygen required for the operation of the POX unit is provided from the oxygen plant (Area 400). Liquid product from the liquefaction section undergoes additional processing in the product upgrading section (Area 600). The entire distillate product is hydrotreated and the heavy naphtha fraction is catalytically reformed in Area 600. The ROSE-SR^{SM} unit is also located in Area 600. Purge gases and sour water from the liquefaction and product upgrading sections are treated in Area 500. The C_4 components are recovered for blending into the gasoline fraction. Ammonia and sulfur are also recovered in Area 500. The main products of the plant are high octane unleaded gasoline, No. 2 diesel fuel, and liquefied petroleum gas (LPG). Areas 700, 800 and 900 provide the plant utilities, product and feed storage, and miscellaneous off-sites.

E. OVERALL MATERIAL BALANCE

Overall plant feed and product rates are provided in *Table 8.4*. The liquefaction section consists of four reactor trains with a total of eight reactors. The coal feed rates to the liquefaction section of the three CTSL design cases are 10,330, 11,210 and 9,800 tons per day, respectively. The capacity of the reactor trains is set by providing the largest diameter reactors that can be shop-fabricated using conventional techniques for thick-walled vessels.

The liquefaction plants which are based on Periods 26 and 43 of POC-01 produce 12,588 and 13,502 BPSD of high octane unleaded gasoline, respectively. The base case plant produces 12,512 BPSD of gasoline. In addition, the plants produce 30,572, 32,792 and 30,450 BPSD of No. 2 Diesel fuel, respectively. The Period 26 plant design generates 381 TPSD of sulfur and 155 TPSD of ammonia. The Period 43 plant design generates 413 TPSD of sulfur and 166 TPSD of ammonia. The base case plant generates significantly less sulfur due to the lower sulfur content of the feed coal. The base case plant design produces 225 TPSD of sulfur and 157 TPSD of ammonia. The sulfur and ammonia by-products provide revenue credits. Although the base case plant feed coal contains more ash, all three plants produce roughly the same quantity of ash that must be disposed. This is due to the higher coal feed rates of the Period 26 and 43 plant designs. A cost is incurred for the disposal of the ash by-product.

The plant utilizes combustion turbine generators to produce all of the electricity required by the plant. As a result, none of the electrical power consumed is purchased. Natural gas is blended with medium BTU gas for the combustion turbines. Therefore, a significant amount of natural gas must be purchased by the plant. The base case requires significantly more natural gas than either the Period 26 or 43 design. The base case requires 110 MMSCFD of natural gas to be purchased. The Period 26 and 43 designs demand 94 and 97 MMSCFD, respectively.

F. PRODUCT QUALITIES

The estimated product qualities of the gasoline and diesel fuel products from the design plant are presented in *Table 8.5*. The Period 26 and 43 plant designs provide slightly higher quality products. The research octane number (RON), Reid vapor pressure (RVP) and octane (R+M)/2 were not predicted for Periods 26 and 43. However, they are anticipated to be the same or higher than the base case.

Gasoline produced from the liquefaction plant is high octane unleaded. The predicted API gravity of the gasoline is 43.6 for Periods 26 and 43 and 41.4 for the base case designs. The octane of the gasoline is 90 (R+M)/2. Catalytic reformer severity governs the octane of the gasoline product. The gasoline product is ideal for blending with lower octane petroleum derived gasoline.

The predicted API gravity of the diesel fuel is 34.7 for the Periods 26 and 43 designs and 32.9 for the base case design. The cetane number of the diesel fuel is anticipated to be greater than 40. In addition, the diesel fuel is essentially sulfur or nitrogen free.

G. HYDROGEN BALANCE

The overall plant hydrogen balance is presented in *Table 8.6*. The hydrogen consumption consists of the hydrogen used in the liquefaction section, used for product upgrading, and purge and solubility losses. The hydrogen consumption for Periods 26 and 43 are 6,603 and 6,437 SCF/B of liquid products, respectively. The hydrogen consumption for the base case is 7,029 SCF/B. The liquefaction section is responsible for roughly 88 % of the hydrogen that is consumed.

Hydrogen is produced via steam reforming of natural gas and partial oxidation (POX) of the solids-containing stream from the solids separation section (ROSE-SR^{SM}). Partial oxidation of the ROSE-SR^{SM} bottoms generates 118 and 141 MMSCFD of hydrogen for Periods 26 and 43. For the base case, 41 MMSCFD of hydrogen is produced by the POX unit. Steam reforming produces 167, 157 and 261 MMSCFD in the Period 26, 43 and base case designs, respectively.

H. THERMAL EFFICIENCY

Table 8.7 presents the thermal efficiency of the design liquefaction plants. Thermal efficiency is defined as the percentage of the energy leaving the plant in the plant products relative to the energy input to the plant.

Energy input includes energy contained in the coal feed, natural gas and purchased electric power. The total energy inputs for Periods 26 and 43 are 365 and 392 GBTU/D. The base case energy input is 373 GBTU/D.

Energy outputs consist of the energy contained in the gasoline, diesel fuel, LPG, sulfur and ammonia products. The outputs contain 268, 284 and 275 GBTU/D for Periods 26 and 43 and the base case, respectively. The resulting thermal efficiencies are 73.4, 72.6 and 73.6 %.

I. UTILITIES SUMMARY

The liquefaction plant utility usage is summarized in *Table 8.8*. All of the utilities are supplied within the grassroots facility with the exception of the purchased natural gas. The summary provides the consumption of electric power, steam, cooling water, process fuel and raw water. The base case uses less electric power and steam than Period 26 and 43. However, the base case design requires more process fuel. All of the plant designs generate roughly the same quantity of raw water.

J. CAPACITIES OF PROCESS UNITS AND OFFSITES

The capacities of the process units and off-sites are provided in *Table 8.9*. The process units consist of Areas 200, 300, 400, 500, 600 and 700. Off-site units consist of Areas 100, 800, 900 and 1000. The partial oxidation unit and related oxygen plant are significantly larger for the Period 26 and 43 designs. The utilities (power distribution, steam generation and cooling water) capacities are relatively the same for all three plant designs. Tankage capacities are similar with the exception of the LPG product. The base case design generates significantly more LPG.

K. LIQUEFACTION PLANT INVESTMENT DETAILS

The equipment specifications used in each liquefaction section design are prorated on the basis of material and energy balance data. Designs of the other areas are based on the latest process and economic information available for all on-site and off-site areas required for a grassroots coal liquefaction facility.

The major equipment costs and plant investment for the liquefaction section of the grassroots liquefaction plant are shown in *Table 8.10*. The capital cost basis is 1991 dollars at a U.S. Gulf coast location. The most economical liquefaction plant design consists of four parallel reactor trains. The capacity of each train is dictated by the maximum size reactors that can be manufactured using conventional techniques. Liquefaction section major equipment consists of pumps, reactors, heaters, exchangers, drums, towers, compressors and hydrogen purification equipment. Major equipment costs are based on vendor input or recent quotations for similar equipment. The major equipment costs for the Period 26 and 43, and base case designs are \$187.3, \$186.1 and \$175.8 MM, respectively.

The total estimated erected cost of the liquefaction plant is the sum of the direct material costs, labor costs, indirect costs and project contingency. The contingency of the liquefaction plant is not shown because it has been applied to the overall plant. The contingency is used to allow for the cost of additional equipment that could be specified in a more detailed design. Commodity materials and labor were determined by using statistical techniques which HRI has developed for the H-Coal and H-Oil processes. Indirect costs are factored from the total direct cost and include field supervision, sales tax, engineering fees, and home office fees. Liquefaction plant investment costs for the Period 26 and 43 and base case designs are \$608.4, \$604.4 and \$571.1 MM, respectively.

L. TOTAL PLANT INVESTMENT SUMMARY

A summary of the total plant investment costs for each design case is provided in *Table 8.11*. The cost of each area of the plant is provided in the table. The total plant investments for the Period 26, Period 43 and base case designs are $2.23, $2.29 and $2.20 x$10^9$, respectively. Home office fees and contingency are roughly 23% of the total plant investment. The partial oxidation and steam reforming units are included in the hydrogen manufacturing area (Area 300).

M. ECONOMIC BASIS

The economic basis is the same as used in the DOE baseline design. The life of the project has been set at 25 years with a tax rate of 34%. The depreciation term is 10 years. A service factor of 0.9 has been chosen which corresponds to roughly 328 days of operation per year. Inflation has been assumed to be 3% and the interest rate fixed at 8%. In addition, the discounted cash flow return on equity has been set at 15%.

N. PRODUCT COST CALCULATION

The product cost calculations for Periods 26 and 43 and the base case are shown in *Table 8.12*. The basis for each operating cost is indicated in the table. For example, the price of Illinois No. 6 coal is $20.50 per ton. The product cost is calculated by adding all of the operating costs, including capital-related costs, subtracting the by-product credits and dividing by the barrels of product that the plant generates. The equivalent crude oil price is obtained by multiplying the product cost by the crude oil equivalent factor. This factor relates the prices of the finished refinery products (gasoline and diesel) to crude oil prices. This accounts for the fact that liquid products from a direct coal liquefaction plant have a greater value than crude oil. Product costs are $38.43, $37.10 and $37.66/B for Periods 26 and 43 and the base case. The corresponding crude oil equivalent factors are 0.842, 0.836 and 0.839. As a result, the equivalent crude oil prices (ECP) are $32.36, $31.02 and $31.58/B, respectively.

O. SENSITIVITY STUDIES

A sensitivity analysis was performed for each plant design to determine the effects of total investment, feed coal, natural gas, and catalyst and chemical costs on the equivalent crude oil price (ECP). The sensitivity of total investment cost on the ECP is presented in *Figure 8.3.* Total investment has the most significant effect on the ECP. A fifteen percent decrease in the total investment reduces the ECP by roughly \$3/B. Sensitivity diagrams for feed coal and natural gas costs are provided in *Figures 8.4 and 8.5.* An increase or decrease in coal or natural gas cost has roughly the same effect on the ECP. A fifteen percent increase in the price of the feed coal or natural gas increases the ECP by roughly \$0.75/B. Reducing the cost of catalyst and chemicals by fifteen percent reduces the ECP by roughly \$0.50/B. The sensitivity of catalyst and chemical costs is shown in *Figure 8.6.*

P. SUMMARY OF RESULTS AND CONCLUSIONS

HRI has completed the economic assessment of the POC-01 CTSL PDU operation. The economic analysis has incorporated the economic bases and financing assumptions recommended by DOE. Costs and utility requirements of plant sections outside the liquefaction section have been factored from the baseline design.

Results from the POC-01 economic assessment confirm the results of prior economic evaluations which were based on bench-scale studies. The product cost for the commercial liquefaction plant designs that are based on POC-01 PDU operation varies from \$31.02 to \$32.36/B. The base case plant design, which is based on bench-scale data, generates products at an ECP of \$31.56/B.

The economic analysis results of Period 43 (Condition 3B) indicates that increasing the space velocity causes a significant decrease in the ECP. A change in space velocity from 19.4 lb/hr/ft^3 in Period 26 to 24.2 lb/hr/ft^3 in Period 43 reduced the ECP 4%, from 32.36 to \$31.02/B.

Sensitivity analysis results show that the largest contributor to the ECP is the capital investment. A fifteen percent decrease in the total investment reduces the ECP by roughly \$3/B. Coal and natural gas costs have only a slight effect on the ECP. A fifteen percent reduction in the cost of the feed coal or natural gas reduces the ECP by only \$0.75/B. The cost of catalyst and chemicals have an even smaller effect on the ECP. The ECP is reduced by roughly \$0.50/B by a fifteen percent decrease in the cost of catalyst and chemicals.

Q. RECOMMENDATIONS

Results of this economic analysis show that operation at higher space velocity (i.e throughput) improves the economics of coal liquefaction. Future experimental work in the POC program should be directed at demonstrating process improvements with the potential to increase space velocity and/or reactor throughput.

TABLE 8.1

DESIGN COAL FEED ANALYSIS

CASE	DESIGN COAL	BASE CASE
Coal Type	Illinois #6	Illinois #6
Mine	Crown II	Burning Star
PROXIMATE ANALYSIS, Wt% DRY COAL		
Volatile Matter	41.19	37.85
Fixed Carbon	48.41	50.65
Ash	10.40	11.50
TOTAL:	100.00	100.00
ULTIMATE ANALYSIS, Wt% DRY COAL		
Carbon	70.28	71.00
Hydrogen	4.73	4.80
Nitrogen	1.33	1.40
Sulfur	4.17	3.20
Oxygen (by difference)	9.09	8.00
Chlorine	0.00	0.10
Ash	10.40	11.50
TOTAL:	100.00	100.00
H/C Atomic Ratio	0.81	0.81
O/C Atomic Ratio	0.10	0.08
SULFUR FORMS, Wt% DRY COAL		
Organic	2.95	2.35
Pyritic	1.21	0.84
Sulfate	0.01	0.01
TOTAL:	4.17	3.20
Heating Value (HHV), BTU/LB Dry Coal	12,650	13,181
Coal Moisture, Wt%	4.00	3.08

TABLE 8.2

DESIGN BASIS COMPARISON

POC-01, PERIOD 26

CASE	PERIOD 26	DESIGN BASIS	BASE CASE
OPERATING CONDITIONS			
Space Velocity, Lb/Hr/Ft3	19.4	19.4	20.0
Recycle Solvent			
Solvent/Coal, Lbs/Lb	1.26	1.26	1.15
Residual Oil, Wt%	5.8	5.8	47.1
First Stage Temperature, °F	765	765	767
Second Stage Temperature, °F	810	810	823
Catalyst Replacement Rate, Lbs/Ton	4.8	4.8	2.7
PROCESS YIELDS, Wt% DRY COAL			
H_2S	2.08	3.55	2.35
NH_3	1.26	1.48	1.30
H_2O	8.61	9.58	9.93
COx	0.06	0.06	0.09
C1-C3	4.88	4.88	7.77
C4-350°F	17.29	16.39	16.42
350-650°F	42.71	40.83	30.70
650-975°F	7.17	7.29	19.82
975+°F	7.71	7.71	2.83
Unconverted Coal	4.52	4.52	4.24
Ash	10.40	10.40	11.50
TOTAL:	106.69	106.69	106.95
PROCESS PERFORMANCE			
Coal Conversion, Wt% MAF	95.0	95.0	95.2
Resid Conversion, Wt% MAF	86.4	86.4	92.0
Total Desulfurization, Wt%	78.6	80.1	69.1
Organic Desulfurization, Wt%		95.6	78.8
Denitrogenation, Wt%	75.1	91.7	76.5
C4-975°F, Wt% Dry Coal	64.8	64.5	66.9
Hydrogen Efficiency	10.0	9.6	9.6

TABLE 8.3

DESIGN BASIS COMPARISON

POC-01, PERIOD 43

CASE	PERIOD 43	DESIGN BASIS	BASE CASE
OPERATING CONDITIONS			
Space Velocity, Lb/Hr/Ft3	24.2	24.2	20.0
Recycle Solvent			
Solvent/Coal, Lbs/Lb	1.31	1.31	1.15
Residual Oil, Wt%	7.0	7.0	47.1
First Stage Temperature, °F	771	771	767
Second Stage Temperature, °F	811	811	823
Catalyst Replacement Rate, Lbs/Ton	4.5	4.5	2.7
PROCESS YIELDS, Wt% DRY COAL			
H2S	1.96	3.52	2.35
NH3	1.16	1.45	1.30
H2O	8.84	9.48	9.93
COx	0.14	0.14	0.09
C1-C3	4.09	4.09	7.77
C4-350°F	14.55	13.39	16.42
350-650°F	45.07	40.45	30.70
650-975°F	4.29	8.69	19.82
975+°F	9.76	9.76	2.83
Unconverted Coal	4.76	4.76	4.24
Ash	10.40	10.40	11.50
TOTAL:	105.02	106.13	106.95
PROCESS PERFORMANCE			
Coal Conversion, Wt% MAF	94.7	94.7	95.2
Resid Conversion, Wt% MAF	83.8	83.8	92.0
Total Desulfurization, Wt%	73.1	79.7	69.1
Organic Desulfurization, Wt%		94.7	78.8
Denitrogenation, Wt%	67.4	89.8	76.5
C4-975°F, Wt% Dry Coal	62.7	62.5	66.9
Hydrogen Efficiency	12.5	10.1	9.6

TABLE 8.4

FEED AND PRODUCT RATES

CASE	PERIOD 26	PERIOD 43	BASE CASE
COAL FEED, TPSD	10,330	11,210	9,800
Purchased Natural Gas, MMSCFD	94	97	110
LIQUID PRODUCTS, BPSD			
Gasoline	12,588	13,502	12,512
Diesel	30,572	32,792	30,450
TOTAL:	43,160	46,294	42,962
Barrels/Ton of Dry Coal	4.18	4.13	4.38
BY-PRODUCTS			
LPG, BPSD	2,258	1,818	3,065
Sulfur, TPSD	381	413	225
Ammonia, TSPD	155	166	157
Ash to Disposal, TPSD	1,087	1,181	1,132

TABLE 8.5

PRODUCT QUALITIES

CASE	PERIOD 26	PERIOD 43	BASE CASE
GASOLINE			
API Gravity, °API	43.6	43.6	41.4
RON (Clear)			95
RVP, Psia			11.5
(R + M) / 2 (Estimated)			90
DIESEL			
API GRAVITY, °API	34.7	34.7	32.9
CETANE NUMBER	>40	>40	>40

TABLE 8.6

PLANT HYDROGEN BALANCE

CASE	PERIOD 26	PERIOD 43	BASE CASE
HYDROGEN CONSUMPTION, MMSCFD			
Liquefaction	260	259	256
Product Upgrading (Net) [1]	15	29	36
Purge and Solubility Losses	10	10	10
TOTAL:	285	298	302
Hydrogen Consumption, SCF/B of Liquid Products	6,603	6,437	7,029
HYDROGEN PRODUCTION, MMSCFD			
Partial Oxidation of Bottoms	118	141	41
Steam Reforming	167	157	261
TOTAL:	285	298	302

[1] - Hydrogen consumed in hydrotreating less hydrogen produced in catalytic reforming.

TABLE 8.7

THERMAL EFFICIENCY

CASE	PERIOD 26	PERIOD 43	BASE CASE
INPUTS, GBTU/D [1]			
Coal	263	286	253
Natural Gas	102	106	120
TOTAL:	365	392	373
OUTPUTS GBTU/D [1]			
Gasoline	70	76	71
Diesel	177	190	178
LPG	15	11	21
Sulfur	3	4	2
Ammonia	3	3	3
TOTAL:	268	284	275
THERMAL EFFICIENCY, %	73.4	72.6	73.6

[1] - G = 10^9 (billions)

TABLE 8.8

UTILITIES SUMMARY

CASE	PERIOD 26	PERIOD 43	BASE CASE
Electric Power (Generated On-Site)	188	198	178
600 Psig Steam, MLb/Hr	252	268	239
Cooling Water, MGPM	147	154	153
Process Fuel, GBTU/D	102	106	120
Raw Water, MGPM	2.2	2.4	2.3

TABLE 8.9

CAPACITIES OF PROCESS UNITS AND OFFSITES

CASE	UNITS	PERIOD 26	PERIOD 43	BASE CASE
PROCESS UNITS				
100 - Coal Preparation	TPSD Dry Coal	10,330	11,210	9,800
200 - Liquefaction	TPSD Dry Coal	10,330	11,210	9,800
300 - Hydrogen Production				
Partial Oxidation	MMSCFD H_2	118	141	41
Steam Reforming	MMSCFD H_2	167	157	261
400 - Oxygen Plant	TPSD Oxygen	1,426	1,733	510
500 - Product Treating				
Acid Gas Removal	TPSD H_2S & CO_2	64	79	45
Sour Water Stripping	TPSD Sour H_2O	13,187	14,147	12,994
Sulfur	TPSD Sulfur	381	413	225
Light Ends	TPSD C_4	130	97	221
600 - Product Upgrading				
Hydrotreating	BPSD Coal Liquid	41,507	43,654	40,095
Catalytic Reforming	BPSD 180-350°F	12,588	13,502	12,512
700 - Utilities				
Power Distribution	MW	188	198	178
Steam Generation	MLb/Hr	252	268	239
Cooling Water	MGPM	147	154	153
800 - Tankage				
Liquid products	BPSD Liquids	43,160	46,294	42,962
Waste Solids Handling	TPSD Solids	1,087	1,181	1,132
LPG	BPSD LPG	2,258	1,818	3,065
900 - General Off-sites	TPSD Dry Coal	10,330	11,210	9,800

TABLE 8.10

LIQUEFACTION PLANT INVESTMENT DETAILS

CASE	PERIOD 26	PERIOD 43	BASE CASE
MAJOR EQUIPMENT COSTS, $M			
Pumps	24,096	25,460	21,741
Reactors	56,280	50,990	53,092
Heaters	14,123	15,046	13,423
Exchangers	17,171	17,896	16,488
Drums, Towers	34,394	35,401	30,153
Compressors	27,291	27,203	27,038
HPU	13,964	14,090	13,901
TOTAL:	187,320	186,093	175,835
PLANT INVESTMENT, $MM			
Equipment & Materials	339.4	337.2	318.6
Labor & Subcontracts	146.9	145.9	137.8
Distributable Indirects	122.2	121.4	114.7
Contingency	0.0	0.0	0.0
LIQUEFACTION PLANT INVESTMENT:	**608.4**	**604.4**	**571.1**

TABLE 8.11

TOTAL PLANT INVESTMENT SUMMARY

CASE	PERIOD 26	PERIOD 43	BASE CASE
AREA INVESTMENT, $MM			
100 - Coal Preparation	47	51	45
200 - Liquefaction	608	604	571
300 - Hydrogen Manufacture	236	245	236
400 - Oxygen Plant	56	64	27
500 - Product Treating	198	192	235
600 - Upgrading & Solids Separation	115	123	131
700 - Utilities	245	254	238
800 - Tankage	121	129	122
900 - General Off-Sites	191	202	185
Sub-Total:	1,818	1,864	1,792
Fee, Contingency	413	423	406
TOTAL PLANT INVESTMENT:	**2,231**	**2,287**	**2,198**
$/BPD of Product	51,700	49,400	51,200

TABLE 8.12

PRODUCT COST CALCULATION

OPERATING COSTS, $MM/Year	PERIOD 26	PERIOD 43	BASE CASE
Coal ($20.50/Ton)	69.56	75.49	66.00
Natural Gas ($2.00/MMBTU)	67.32	69.33	78.95
Water ($0.10/MGal)	0.37	0.40	0.38
Catalyst & Chemicals	53.53	53.99	38.93
Ash, Waste Disposal ($5.00/Ton)	1.79	1.94	1.86
Labor	23.07	23.07	23.07
Maintenance	20.67	20.67	20.67
Capital-Related	336.29	345.74	331.59
TOTAL:	**572.60**	**590.62**	**561.45**
BYPRODUCT CREDITS, $MM/Year			
LPG ($9.90/B)	11.61	8.98	17.86
Sulfur ($80/Ton)	10.00	10.85	6.19
Ammonia ($120/Ton)	6.11	6.55	5.91
TOTAL:	**27.72**	**26.38**	**29.96**
PRODUCT COST	**544.88**	**564.24**	**531.49**
Product Cost, $/B	38.43	37.10	37.66
Crude Oil Equivalent Factor	0.842	0.836	0.839
Equivalent Crude Oil Price, $/B	**32.36**	**31.02**	**31.58**

FIGURE 8.1

BLOCK FLOW DIAGRAM OF MAJOR PROCESSING AREAS

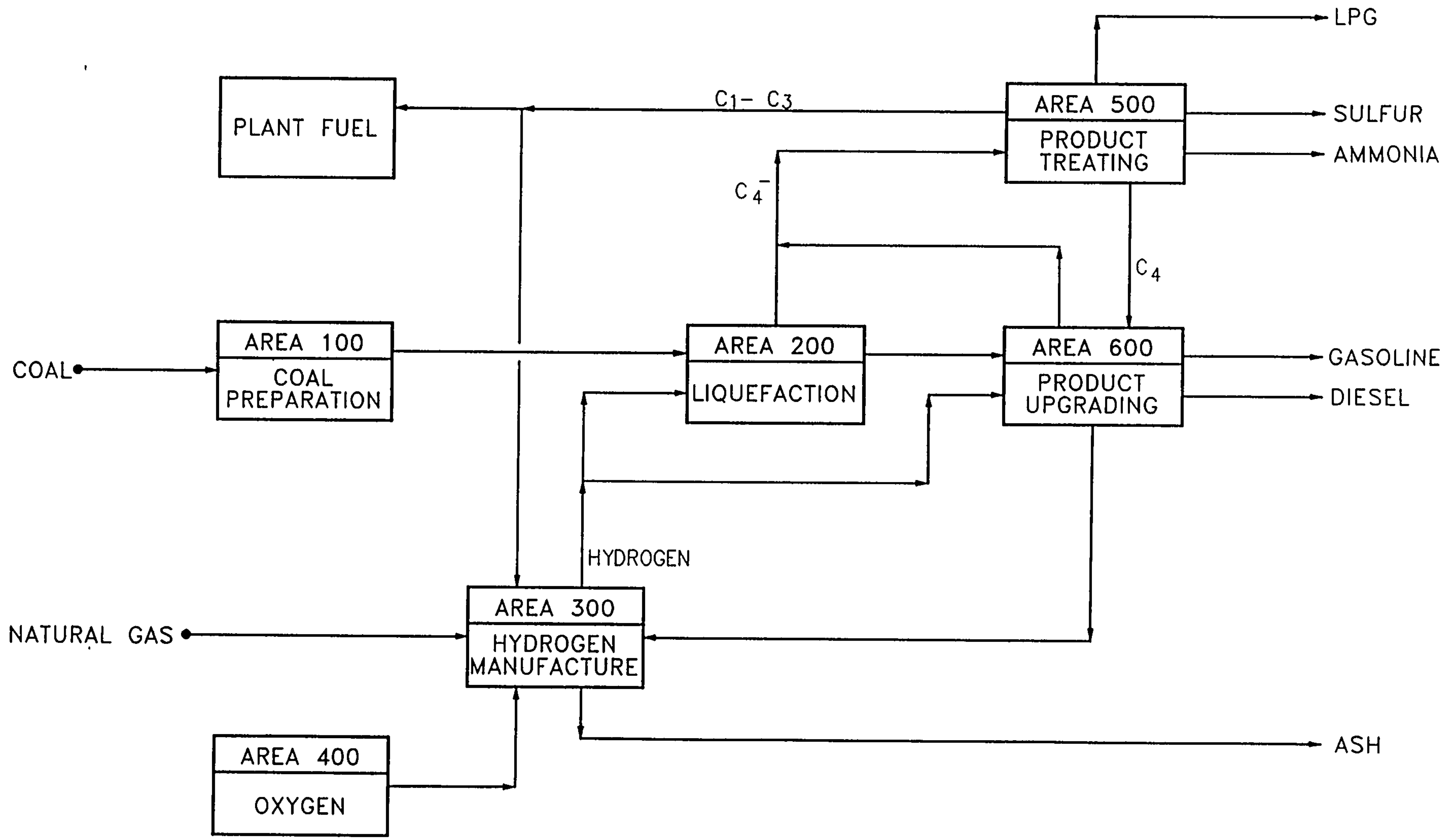


FIGURE 8.2

CATALYTIC TWO-STAGE COAL LIQUEFACTION (CTSL™) PROCESS

SIMPLIFIED FLOW PLAN

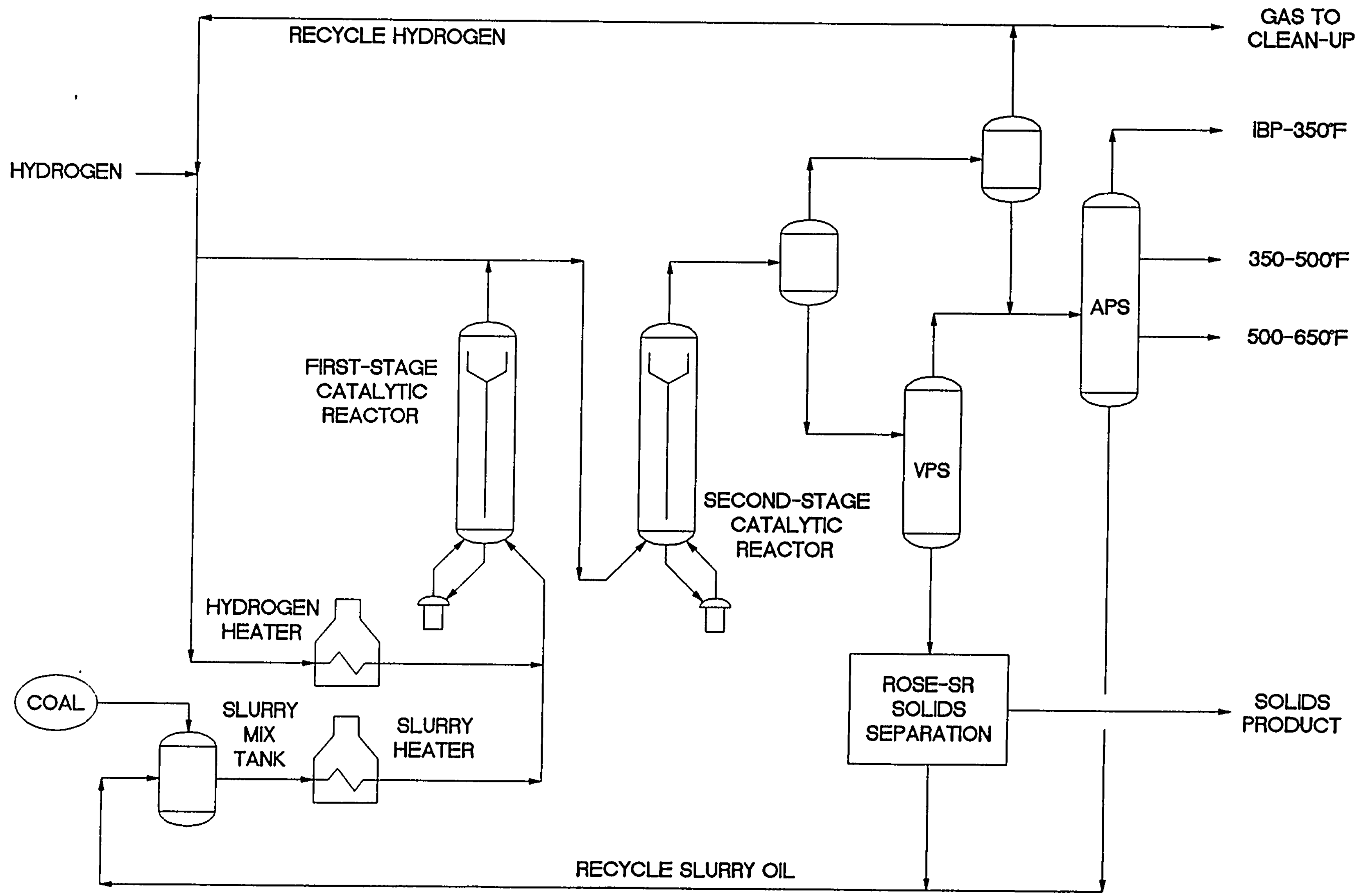


FIGURE 8.3

SENSITIVITY ANALYSIS
EFFECT OF TOTAL INVESTMENT

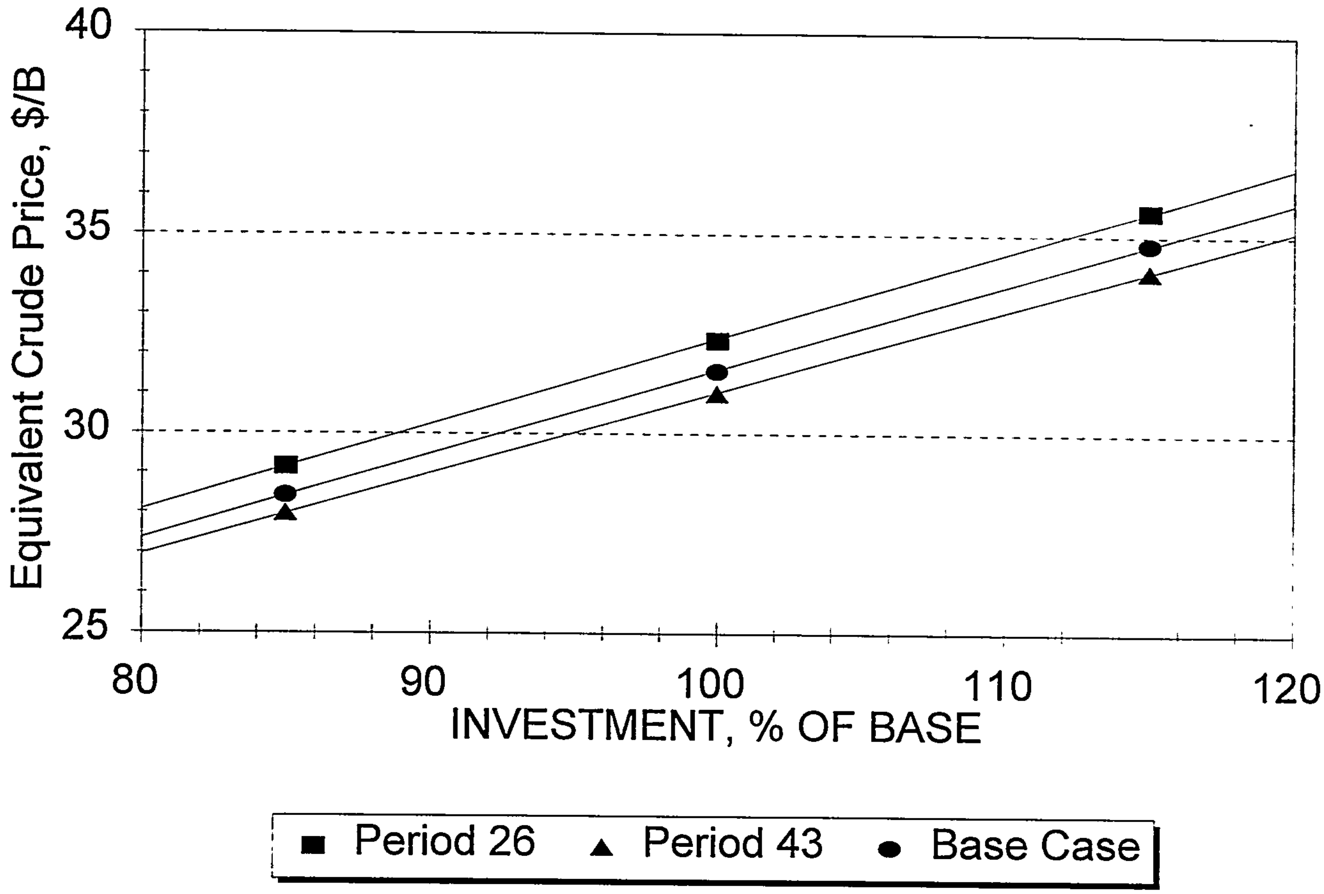


FIGURE 8.4

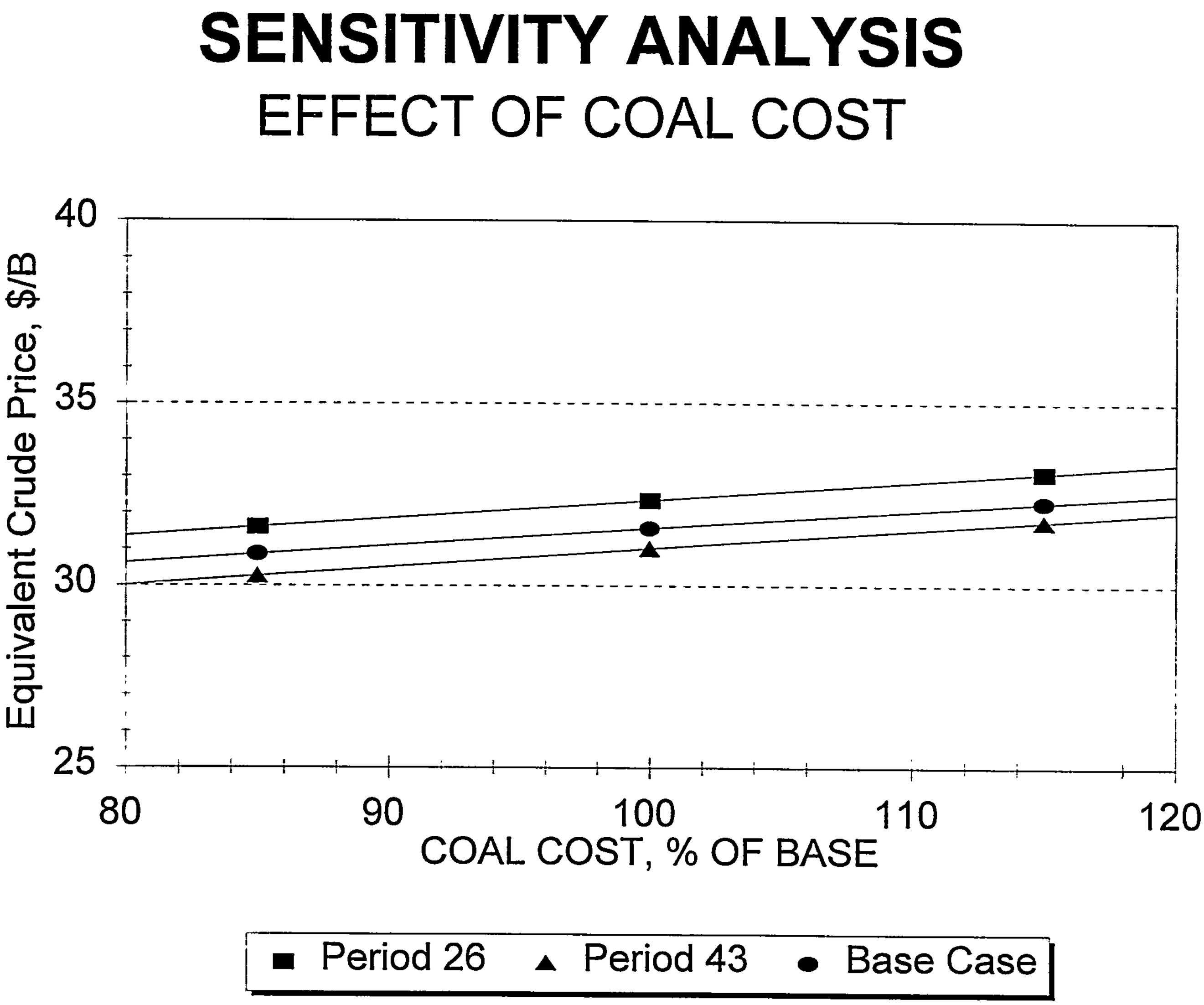
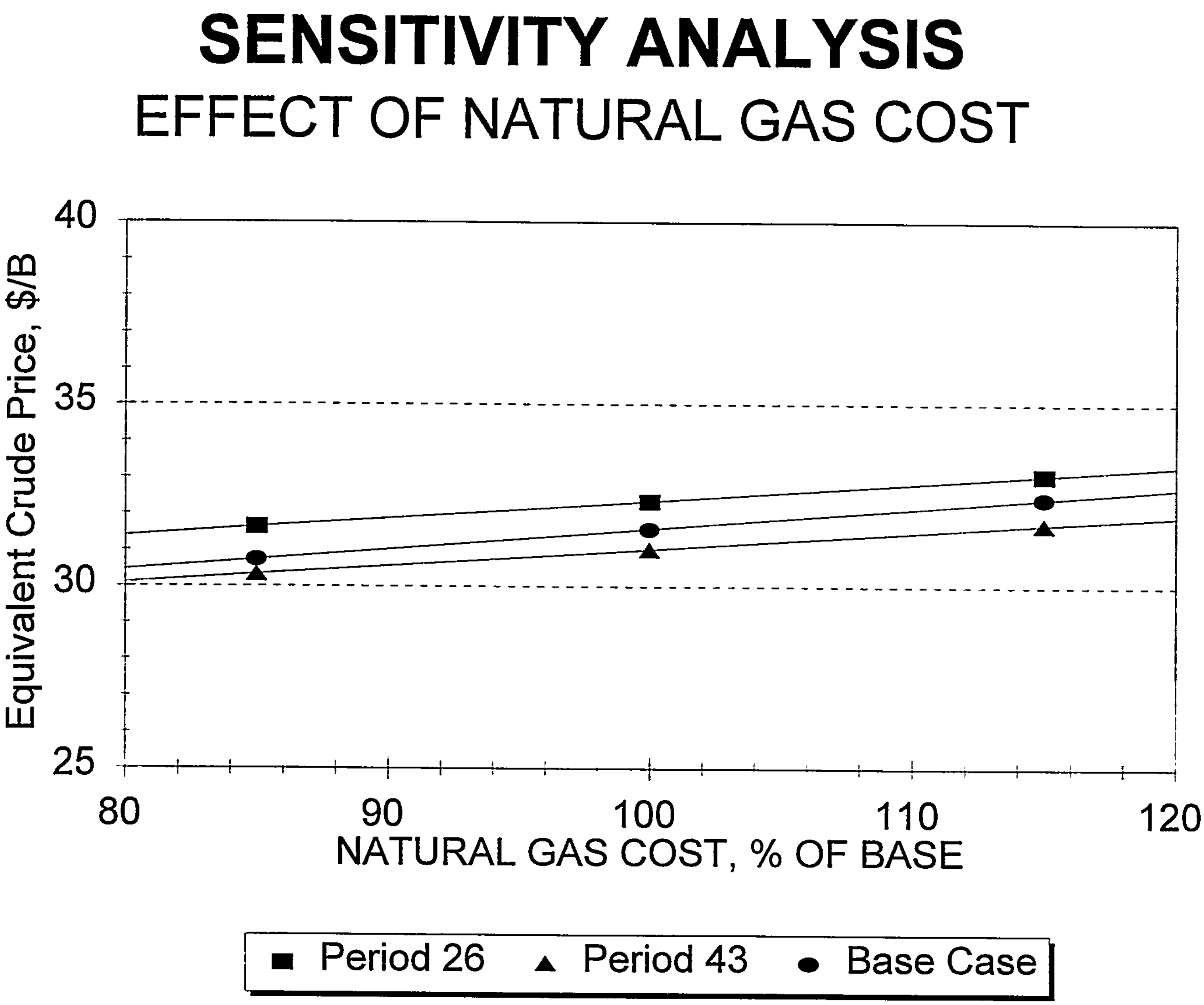
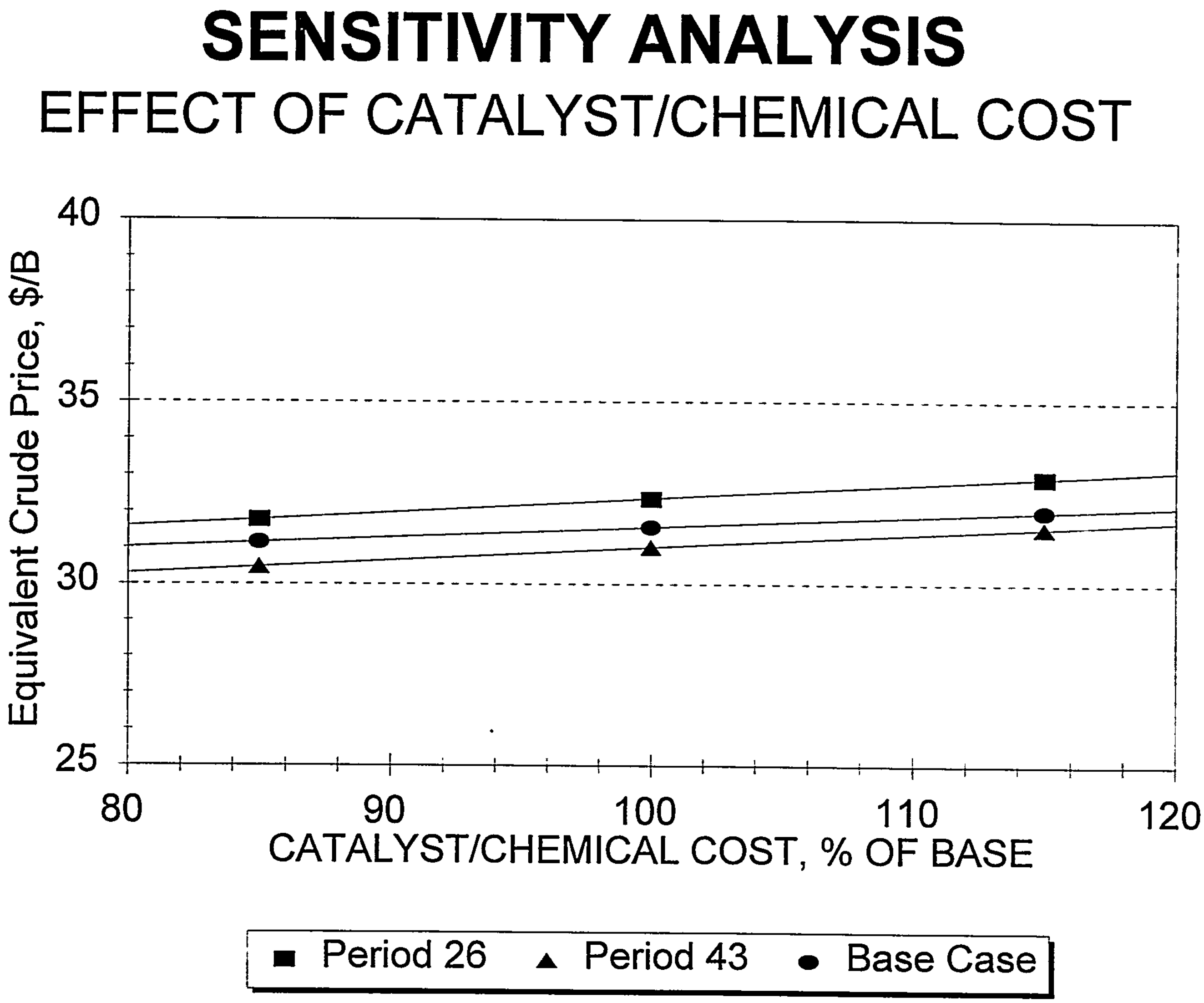

SENSITIVITY ANALYSIS
EFFECT OF COAL COST
Equivalent Crude Price, $/B
40
35
30
25
80
90
100
110
120
COAL COST, % OF BASE
Period 26
Period 43
Base Case

FIGURE 8.5



SENSITIVITY ANALYSIS
EFFECT OF NATURAL GAS COST
40
35
30
25
Equivalent Crude Price, $/B
80
90
100
110
120
NATURAL GAS COST, % OF BASE
Period 26
Period 43
Base Case

FIGURE 8.6


SENSITIVITY ANALYSIS
EFFECT OF CATALYST/CHEMICAL COST
Equivalent Crude Price, $/B
40
35
30
25
80
90
100
110
120
CATALYST/CHEMICAL COST, % OF BASE
Period 26
Period 43
Base Case

SECTION IX

SAMPLES/MATERIAL TESTING

A. EXTERNAL SAMPLES

Among the several objectives of PDU run POC-01, one was to collect samples of different process streams for both analyses and end-use property inspections of various internal process streams as well as the end-product distillate liquids. Several requests for these POC-01 samples were made by other U.S. DOE contractors; Consol, Inc. and Bechtel, Inc. were among the most important sample recipients. *Table 9.1* lists all the daily and special samples collected during the PDU run. The daily samples of gaseous, liquids, and solid products and various internal streams were analyzed in house at HRI to determine process yields and product quality. *Figure 9.1* shows the locations of the sampling points on the POC-01 flow diagram (*Table 9.1* lists all the sampling during POC-01). A list of all external samples and their recipients is given in *Table 9.2*. External samples mainly consisted of recycle oil (O-43 oil), naphtha stabilizer bottoms (N-5), O-13 reactor flash vessel bottoms, and make-up oil (Tank 4 oil) for the PDU run. The special samples for Consol, Inc. consisted of a number of different process streams and were collected only during the full work-up periods of POC-01. These include Periods 9, 19, 26, 43, and 57 (*Table 9.3*).

The special sample for Bechtel, Inc. was a 3000 gallons distillate sample collected in three different compartments of a tank truck from the equilibrated Periods (41 through 50) of POC-01. This sample was collected for the end-use characterization and upgrading to be conducted for Bechtel by Southwest Research Institute in Texas. The collection history of this distillate sample, in terms of run conditions, process mass balance, and distillate yields is given in *Table 9.4*. Run conditions were very consistent throughout, although process performance degraded, especially beyond Period 48, mainly because catalyst replacement was not operable after Period 48. Failure to effect daily catalyst replacement affected the properties of the distillates collected. As shown in *Table 9.5*, the properties of the distillates in terms of boiling range and heteroatom contents are very consistent until Period 49. The distillate sample, collected in the rear compartment of the tank truck during periods after 48-49 shows a higher heteroatom content.

B. MATERIAL TESTING

MATERIAL TESTING

During POC-01 a cooperative material testing program between Oak Ridge National Laboratory (ORNL) and HRI was implemented. Six sets of corrosion coupons were supplied by ORNL and installed at selected locations in the PDU. These locations included reactors, hydrotreater, hot separator, atmospheric and vacuum stills. Due to a scheduling problem, the set of coupons intended for the hot separator was not installed.

Each set consisted of 5 to 7 individual coupons of different materials. The coupon material and location for each set are given in Table 9.6. The corresponding exposure times are summarized in Table 9.7. The Hydrotreater and Atmospheric Still coupons, which were placed in the unit in on October 18, 1993, had the longest exposure time of 58 days. The second stage reactor coupons had an intermediate time of 48 days, while the remaining coupons were exposed to process fluids in the range of 20 to 22 days.

Up to this point, five sets of coupons have been removed. The analysis of the first set (Hydrotreater) has been completed, and results are reported in the table below:

Material	Corrosion Rate	Corrosion Rate
	mm/yr	mil/yr
2 1/4 Cr - 1 Mo Steel	1.58	62.3
Modified 9 Cr - 1 Mo Steel	1.14	44.8
316L Stainless Steel	0.19	7.5
Incoloy 825	0.07	2.7
321 Stainless Steel	0.03	1.0
347 Stainless Steel	<0.03	<0.1
304L Stainless Steel	<0.03	<0.1
Fe_3Al	<0.03	<0.1

Note: Sample exposed during full temperature operation for 26 days on coal and 16 days on oil.

In calculating the corrosion rate a correction was made to the operating days during which the hydrotreater was bypassed. According to ORNL, the observed corrosion rates followed a pattern typical of metals exposed in sulfidizing coal liquefaction environments where the corrosion rate is generally a function of chromium content. Examination of the second set of coupons is currently underway at ORNL.

TABLE 9.1

POC PDU Run 1 : Sampling

*DAILY/SPECIAL SAMPLES***

Sample Point	Description	Typical Amounts
SP-1	Vent Gas	Flow Bottle
SP-2	Bottom Gas	Flow Bottle
SP-3	N-5 Bottoms: NSBs	1/2 gallon
SP-4	N-2 Bottoms: ASBs	1/2 gallon
SP-5	N-3 Ovhd: VSOH	1 quart
SP-25	O-65: Rose-SR DAO	1 quart
SP-26	O-67: Rose-SR Solvent	1 pint
SP-24	P-3: COT (m/u + vso)	1 pint
SP-14	O-44: Sour Water	1 pint
SP-15	P-4, SMT Condensate	1 pint
SP-6	N-3 Bottoms: VSBs	1 quart
SP-7	P-4: SMT Feed Slurry	1 pint
SP-9	O-46: RLFV (O-13) Bottoms	1 quart
SP-11	O-43: Recycle Oil	1 quart
SP-12	K-1 Slurry	Whole Sample
SP-33	O-61: Rose-SR Feed	1 quart
SP-16	P-2: Feed Coal	100 gm
SP-27A/B	O-63A/B: Rose-SR Bottoms	1 quart
SP-17	K-1 Catalyst Withdrawal	Whole Sample
SP-18	K-2 Catalyst Withdrawal	Whole Sample

* All the samples are collected during sub-period B nearing completion of an Operating Period except for K-1 reactor slurry and catalyst withdrawals

**Daily and some Special samples are for the HRI Internal Analyses; while most of the Special Samples (Varying Amounts) were taken for External Recepients (other related DOE Programs)

TABLE 9.2

The Summary of External Samples from POC-01

Sample Recepient	Sample Type	Amount	Special Instructions	Status
Alberta Res. Council Canada	Recycle oil [O-43 oil]	660 lbs (2 drums) [Composite]	Periods 41 through 51	Two Drums (85 gallons total) sent
Bechtel [for SwRI]	C5-750F Net Distillates	<=2500 Gallons	Net Distillates* (NSB+ASB) Condition 3 & 4	> 3000 gallons in a Truck Under N2 (Shipped)
Bechtel [for SwRI]	C5-750F Net Distillates	Two Gallon	Condition 3 & 4	Already Sent
CAER, UK [For B.H. Davis]	IBP-650 F Product	20 Gallons	Period 48	Already Sent
CAER, UK [G. Kimber]	O-63 Rose BTMS O-65 DAO N-2 ASBs N-3 VSBs	One Lb One Lb One Lb One Lb	Period 57 Period 57 Period 57 Period 57	Shipped do do do
CeraMem Corp.	O-13 Bottoms	40 Gallons	Periods 40-57	Shipped
	N-3 Bottoms	50 Gallons	End of Run	Shipped
Consol, Inc.	A SEPARATE TABLE ATTACHED		Period 9, 19, 26, 43, and 57 samples sent.	
PETC [For Dick Lett]	Tank 4 Material S/U Oil:L-800 (Tank 5 Material)	8 drums 5 drums	End of Run Period 4	Shipped Already Sent
	C5-750F Net Distillates	Two drums	Periods 43-50	Shipped
UOP, Inc. [H.B. Gala]	IBP-650 F Product	10 Gallons	Period 49	Already Sent
Sandia Natl Labs. [Steve Lott]	VSOH	Two Gallons	Period 43-57	Shipped

TABLE 9.3

Special POC-01 Stream Samples for the Consol, Inc.

Samples**	Vessel/s	Amounts	Which Periods
Distillates			
N-5 Bottoms	D-5	100 gms	9, 19, 26, 43, and 57
N-2 Bottoms	D-2	100 gms	9, 19, 26, 43, and 57
N-3 Overheads	D-4	100 gms	9, 19, 26, 43, and 57
Recycle oils			
O-43 oil	O-43	1 gallon	9, 19, 26, 43, and 57
	O-43	2 drums	Composite from Periods 52 through 58
COT Oil	P-3	1 gallon	9, 19, 26, 43, and 57
	P-3	2 drums	Composite from Periods 52 through 58
O-13 Bottoms			
O-46 material	O-46	750 gms	1,2........58 (Each Run Period)
K-1 Reactor Sample	O-71	300 gms	Wheneven Taken
Residues			
N-3 Bottoms	O-60	100 gms	9
Rose Streams			
Rose solids	O-63A/B	100 gms	19, 26, 43, and 57
Rose Feed	O-60/61	100 gms	19, 26, 43, and 57
K-1 Catalyst	O-16	250 gms	9, 19, 26, 43, and 57
	O-16	2.5 lbs	Composite from Periods 1 through 10
K-2 Catalyst	O-34	250 gms	9, 19, 26, 43, and 57
	O-34	2.5 lbs	Composite from Periods 1 through 10
Feed coal	P-2	0.5 lbs	9, 19, 26, 43, and 57
Misc. Oils			
S/U or M/U Oil	Tank 4	1 Quart	9
Rose-DAO	O-65	500 gms	19, 26, 43, and 57

TABLE 9.4

COLLECTION HISTORY OF NET DISTILLATE SAMPLE FOR END-USE

Run Condition	3B	3B	3B	4A	4A	4B	4B
Period No.	41	42	43	47	48	49	50
Coal Sp. Vel., Lb/hr/ft	24.1	25.3	25.3	25.1	25.7	26.6	27.9
Solvent/Coal	1.2	1.2	1.2	1.15	1.15	1.1	1.1
Mass Balance %	97.2	94.3	97.8	100.3	99.8	97.6	100.9
K-1 Temp, F	771	771	771	777	777	774	773
K-2 Temp, F	810	810	810	811	812	812	812
Norm. NSB Yield W% MAF Coal	64.8	68.1	63.7	63	58.7	57.8	53.3

TABLE 9.5

INSPECTION OF NSB DISTILLATE SAMPLE FOR END-USE

Period No.	43	47	49	Trailor Sample Compartment FRONT	MIDDLE	REAR
API Gravity	32.5	33.2	32.5	32.9	32.7	31.8
IBP, F	133	127	138	145	135	136
5 V%	205	208	214	212	195	206
10	239	237	244	245	225	235
20	302	291	299	309	276	287
30	375	348	358	371	344	344
40	439	409	412	430	403	398
50	485	465	458	479	453	447
60	526	505	498	518	503	491
70	555	543	534	550	540	528
80	584	577	568	582	574	562
90	620	617	601	620	615	599
95 V%	647	651	631	650	645	626
EP, F	674	672	662	670	673	670
W%						
IBP-350 F	23.1		25.7	23.8	27.8	27.2
350-550 F	41.5		46.5	43.8	43.2	45.9
550-650 F	29.4		23.1	26.3	23.7	22.4
650 F+	5.6		4.2	5.7	5	4.1
W%						
Carbon	86.5		86.3	86.95	86.96	86.95
Hydrogen	12.2		12.29	12.43	12.38	12.08
Nitrogen	0.058		0.084	0.0582	0.0717	0.122
Sulfur	0.034		0.032	0.0329	0.033	0.0507

TABLE 9.6

POC-01 Corrosion Coupon Materials

Coupon Location	Coupon Materials
Reactors (1)	a. 2 1/4 Cr - 1 Mo Steel b. 9 Cr - 1 Mo Steel c. 321 Stainless Steel d. Al_2O_3 Plasma Spray Coating - A e. Al_2O_3 Plasma Spray Coating - B
Hydrotreater & Hot Separator	a. 2 1/4 Cr - 1 Mo Steel b. Modified 9 Cr - 1 Mo Steel c. 304L Stainless Steel d. 316L Stainless Steel e. 347 Stainless Steel f. Incoloy 825 g. Fe_3Al
Atmospheric Still & Vacuum Still	a. Carbon Steel b. 1 1/4 Cr - Mo Steel c. 2 1/4 Cr - 1 Mo Steel d. 5 Cr - Mo Steel e. 7 Cr - 1 Mo Steel f. Modified 9 Cr - 1 Mo Steel

(1) Reactor corrosion coupons provided by Mitsui SRC Development Co.

TABLE 9.7

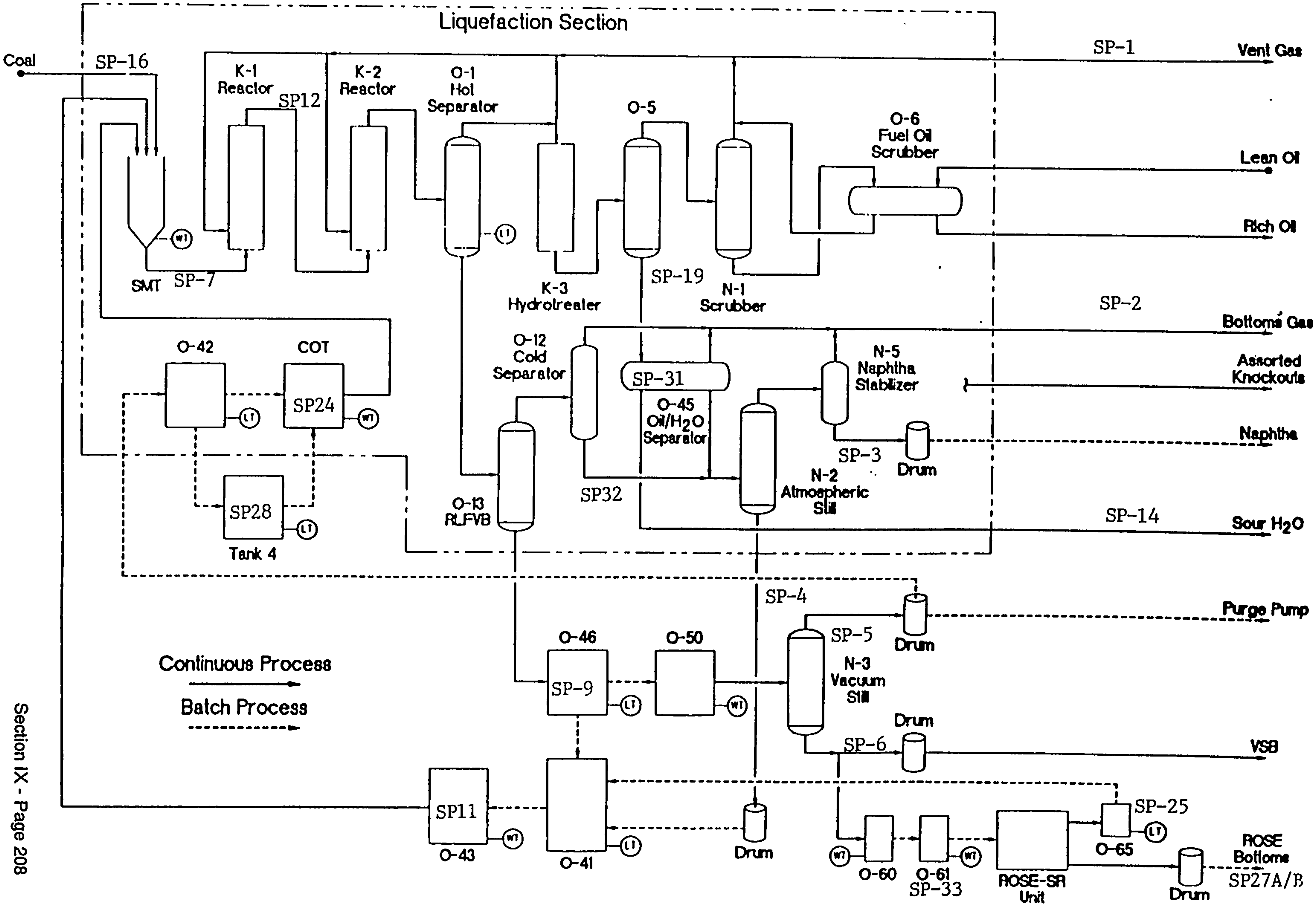
POC-01 Corrosion Coupon Exposure Time

Coupon Location	Date Installed	Operating Days on Coal	Operating Days on Oil
First Stage Reactor - below ebullating cup	01/25/94	20	5
Second Stage Reactor - below ebullating cup	11/24/93	48	18
Hydrotreater - at the bottom section	10/18/93	58 (26)	28 (16)
Hot Separator - in vapor zone	Not installed	0	0
Atmospheric Still - below the packing in the reboiler	10/18/93	58	28
Atmospheric Still - atop the packing near the condenser	10/18/93	58	28
Vacuum Still - in condenser	01/12/94	22	9

Note: Values in bracket are on-line days with vessel at full operating temperature.

260-04 MATERIAL BALANCE FLOW DIAGRAM

FIGURE 9.1



SECTION X

REFERENCES

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2. A.G. Comolli et al., "Catalytic Two-Stage Coal Liquefaction, CTSL-Proof-of-Concept and Developments", a Paper Presented at The NEDO 1994 Coal Liquefaction and Materials for Coal Liquefaction Joint Technical Meeting in Tokyo, Japan, January 1994.

3. "Southern Electric International, Inc., Technical Progress Report", Run 257 with Illinois No. 6 Coal", DOE/PC/50041-121, March 1991.

4. "Final Report on Baseline and Improved Baseline Designs", Bechtel Corporation, under Contract No. DE-AC22 90PC89857, March 1993.

APPENDIX A

Definition and Nomenclature

APPENDIX A

DEFINITION AND NOMENCLATURE

Terminologies, that are used in this report, are defined in the following section:

A. Major Process Equipment

Symbol	Description
L-1	Fresh Feed Heater
L-2	Recycle Gas Heater
K-1	First Stage Reactor
K-2	Second Stage Reactor
K-3	In-line Hydrotreater
N-1	Scrubber
N-2	Atmospheric Tower
N-3	Vacuum Still
N-5	Naphtha Stabilizer Column
O-1	Hot Separator
O-5	Reactor Overheads Separator
O-12	Reactor Liquid Flash Drum
O-13	Reactor Liquid Flash Drum
O-40	Purge Oil Tank
O-41	Recycle Holding Tank
O-42	Flush/Purge Oil Storage
O-43	Recycle Weigh Drum
O-46	O-13 Liquid Surge Drum
O-47	Filter Feed Drum
O-48	Filtrate Receiver
O-50	N-3 Feed Accumulator
O-60	VSB Holding Tank
O-61	Settler Feed Tank
O-63	ROSE Residues Receiver
O-65	Recycle Oil Receiver
P-1	Coal Day Hopper
P-2	Coal Feed Hopper
P-3	Clean Oil Tank
P-4	Slurry Oil Tank

NOMENCLATURE:

Normalization Factor — A factor used to normalized the raw material balance data and is defined as:

$$\frac{100}{\mathit{Wt\%\ Mass\ Recovery}}$$

Normalized yields is equal to the product of net yield multipled by the normalization factor.

Coal Conversion — The conversion of coal into gases, water and quinoline soluble liquid products.

$$100 \times \left[1 - \frac{\mathit{w\%\ Quinoline\ Insoluble\ Organic\ in\ Product}}{\mathit{w\%\ M.A.F.\ coal\ in\ Feed}}\right]$$

524°C+ Resid Conversion: The Conversion of coal and 524°C+ resid into gases and 524°C- distillates

$$100\left[1 - \frac{\mathit{W\%\ 524^{\circ}C{+}\ in\ Product}}{\mathit{W\%\ 524^{\circ}C{+}\ in\ Feed}}\right]$$

Hydrodesulfurization: — The removal of organic sulfur from the net liquid products.

$$100\left[1 - \frac{\mathit{W\%\ Sulfur\ in\ Liquid\ Product}}{\mathit{W\%\ Sulfur\ in\ Total\ Feed}}\right]$$

Hydrodenitrogenation: — The removal of nitrogen from the net liquid products.

$$\text{Hydrodenitrogenation} = 100 \times \left[- \frac{\text{W\% Nitrogen in Liquid and Solid}}{\text{W\% Nitrogen in Total Feed}} \right]$$

Organic Rejection: Rejection of organic matter in ROSE Bottoms or Filter Cake.

$$100 \times \left[\frac{\text{W\% Organic in Ash Reject x Wt. of Adjusted Ash Reject}}{\text{Wt. of Moisture-Ash-Free Coal}} \right]$$

APPENDIX B

Material Balance Methodology

APPENDIX B

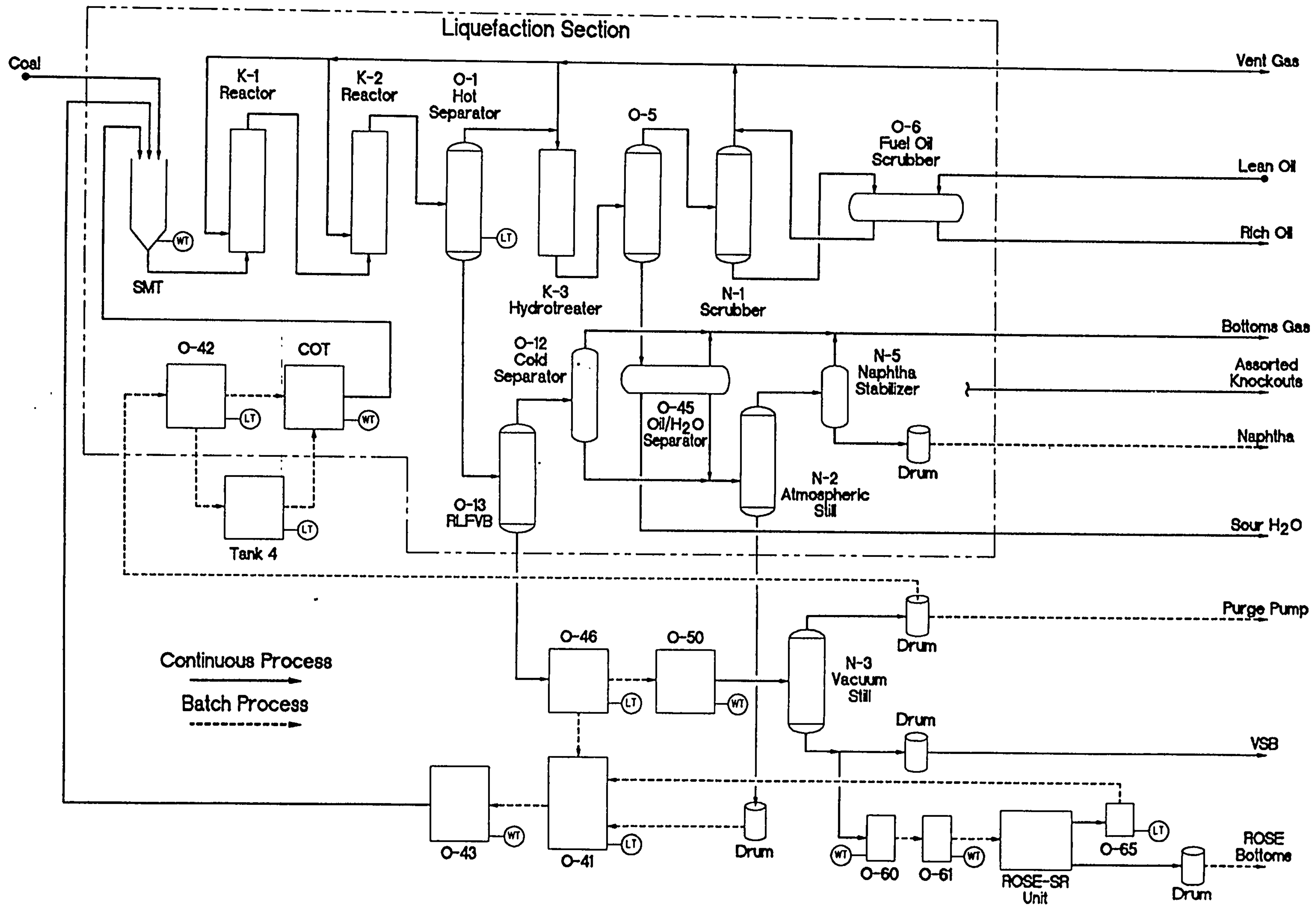
MATERIAL BALANCE METHODOLOGY

The material balance for this run was calculated in two different ways, as an overall unit balance around the entire process and as a liquefaction section balance which is exclusive of the solid separation equipment. As can be seen in Figure B-1, the overall balance includes the various feeds and all the final products, which are taken only after the internal recycle is accounted for. This gives a total balance around all the equipment including whichever solid separation system is being used. This includes a number of batch transfer operations (ie. from O-46 to O-50, from O-41 to O-43) which makes the inventory changes in these vessels critical to a tight material balance closure.

The liquefaction balance is performed exclusive of the solid separation equipment. As can be seen from the figure, this balance stops at the O-46 vessel which is the Reactor Liquid Flash Vessel bottoms receiver. From this point on it is decided how this material is recycled. During ashy-recycle mode a portion of this stream is fed to the O-41 vessel which is part of the recycle oil system. This stream can also be feed directly to the filter system or the Vacuum Still feed tank, through the Vacuum Still and the bottoms then routed to the ROSE-SR^{SM} unit. Due to all the various ways that material in the O-46 vessel can be routed, this was chosen as the cut-off point for the liquefaction balance. Results determined at this point would be independent of which solid separation system is actually being used (except as the quality of the recycle solvent from the various solid separation systems would vary and effect the performance in the reactors). The liquefaction section material balance is the one that is used to determine all the process performance and the normalized yield calculations.

FIGURE B.1

260-04 MATERIAL BALANCE FLOW DIAGRAM



APPENDIX C

Material Balance Data

POC-01 (RUN 260-04) MATERIAL BALANCE
**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	L/O	L/O	L/O	L/O	L/O	L/O	L/O	L/O
Period	01T	02T	03T	04T	05T	06T	07T	08T
Period Start Date	10/29/93	10/30/93	10/31/93	11/01/93	11/02/93	11/07/93	11/08/93	11/09/93
Period Start Time	04:00	04:00	04:00	04:00	04:00	16:00	04:00	04:00
Period Duration Hours	24	24	24	24	12	24	24	24
Solids Separation Type	VAC STIL	VAC STIL	VAC STIL	VAC STIL	VAC STIL	VAC STIL	VAC STIL	VAC STIL
STREAMS IN, KGS								

Coal Feed Wet (Less Sample)	904.8	1220.0	1831.2	1747.6	883.1	799.5	1749.8	1895.3
Make-Up Oil to Mix Tank	3035.1	1774.9	946.2	1046.0	636.8	43.1	703.8	670.5
Mix Tank Inventory Loss	-262.6	275.8	-6.8	79.8	-83.5	-8.2	60.3	-56.2
Seal Oil to Ebullating Pumps	36.7	41.4	54.0	53.1	20.9	22.6	47.3	45.2
Make-Up Oil to Purge Pumps	0.0	57.6	0.0	0.0	0.0	117.6	0.0	0.0
Water Injected to O-1	293.0	441.5	445.9	427.3	210.0	318.6	669.4	690.0
Fresh Hydrogen Feed	130.3	147.1	152.5	145.2	70.3	95.3	133.3	102.8
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	8.7	0.0	0.0
Make-Up Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	4137.2	3958.2	3422.9	3499.0	1737.7	1397.2	3363.9	3347.6
STREAMS OUT, KGS								

Vent Gas (Dry & N2-Free)	42.5	53.1	54.5	49.2	26.7	12.3	45.4	50.1
Bottoms Flash Gas (Dry & N2-Free)	135.9	126.7	189.8	113.7	77.3	23.6	142.2	197.2
Mix Tank Vent Drain	0.8	0.0	0.0	0.0	0.0	0.9	0.4	0.6
Unit Knockouts	0.0	5.0	5.3	124.2	7.3	0.9	4.3	4.0
Naphtha Stabilizer Bottoms	1344.9	1521.7	1406.5	1485.4	766.6	282.0	1268.2	1723.0
Atmospheric Still Bottoms Product	0.8	1.8	47.1	2.6	0.4	1.2	1.6	1.5
Separated Water (Plus Water in Gases)	514.4	574.6	675.6	652.1	218.6	235.6	767.5	810.2
Vacuum Still Overhead Product	2.4	1.5	1.8	1.8	0.5	1.5	1.7	1.8
Vacuum Still Bottoms Product	0.0	631.0	895.0	884.1	474.5	35.1	1396.1	509.3
ROSE Unit DAO Product	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Recycle Oil Net Inv. Change	199.2	273.9	245.4	-19.1	-54.8	33.8	99.0	-6.9
Vacuum Still Feed Tank Inv. Change	347.0	-105.7	-97.5	219.1	134.7	31.3	-96.2	8.2
RLFV Bottoms Holding Tank Inv. Change	99.4	386.7	-39.9	-170.0	-77.3	195.2	-311.2	-112.9
TOTAL PRODUCTS:	2687.3	3470.4	3383.7	3343.1	1574.4	853.3	3319.1	3186.0
OVERALL UNIT MATERIAL RECOVERY, W%	65.0	87.7	98.9	95.5	90.6	61.1	98.7	95.2

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition Period	L/O 01T	L/O 02T	L/O 03T	L/O 04T	L/O 05T	L/O 06T	L/O 07T	L/O 08T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	904.8	1220.0	1831.2	1747.6	883.1	799.5	1749.8	1895.3
Oil Streams to the SMT								
Recycle to SMT	753.4	1727.7	1839.8	1357.1	804.2	1761.3	2439.9	2456.6
Make-Up Oil to SMT	3035.1	1774.9	946.2	1046.0	636.8	43.1	703.8	670.5
VSOH recycled to SMT	189.1	0.0	202.8	330.7	127.5	0.0	172.5	135.5
Mix Tank Inventory Loss	-262.6	275.8	-6.8	79.8	-83.5	-8.2	60.3	-56.2
Seal Oil to Ebullating Pumps	36.7	41.4	54.0	53.1	20.9	22.6	47.3	45.2
VSO to Purge Pumps	132.0	223.5	265.8	191.4	77.6	35.9	382.1	279.7
Make Up Oil to Purge Pumps	0.0	57.6	0.0	0.0	0.0	117.6	0.0	0.0
Water Injected to O-1	293.0	441.5	445.9	427.3	210.0	318.6	669.4	690.0
Fresh Hydrogen Feed	130.3	147.1	152.5	145.2	70.3	95.3	133.3	[illegible]
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	8.7	0.0	0.0
TOTAL FEED:	5211.7	5909.4	5731.2	5378.2	2747.0	3194.4	6358.4	[illegible]
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	42.5	53.1	54.5	49.2	26.7	12.3	45.4	50.1
Bottoms Flash Gas (Dry & N2-Free)	135.9	126.7	189.8	113.7	77.3	23.6	142.2	[illegible]
Mix Tank Vent Drain	0.8	0.0	0.0	0.0	0.0	0.9	0.4	0.6
Unit Knockouts	0.0	5.0	5.3	124.2	7.3	0.9	4.3	4.0
Naphtha Stabilizer Bottoms	1344.9	1521.7	1406.5	1485.4	766.6	282.0	1268.2	1723.0
Atmospheric Still Bottoms	141.4	343.8	531.5	554.2	215.8	303.7	441.1	[illegible]
Separated Water (Plus Water in Gases)	514.4	574.6	675.6	652.1	218.6	235.6	767.5	[illegible]
Reactor Liquid Flash Vessel Bottoms	1692.3	3475.9	2807.5	2248.0	1273.7	2932.5	3655.0	[illegible]
TOTAL PRODUCTS:	3872.3	6100.8	5670.8	5226.7	2586.0	3791.4	6324.2	6060.1
LIQUEFACTION SECTION RECOVERY, W%	74.3	103.2	98.9	97.2	94.1	118.7	99.5	97.4
SOLVENT TO COAL (MF) RATIO	4.49	2.93	1.66	1.59	1.81	2.31	1.94	1.78

Condition	L/O	L/O	L/O	L/O	L/O	L/O	L/O	L/O
Period	01T	02T	03T	04T	05T	06T	07T	08T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to Vacuum Still	437.7	1550.4	1234.2	1310.4	595.6	1221.1	1636.1	841.9
STREAMS OUT, KGS								
Vacuum Still Overhead Product	323.5	225.1	470.4	523.9	205.6	37.4	556.4	417.0
Vacuum Still Bottoms Product	0.0	631.0	895.0	884.1	474.5	35.1	1396.1	509.3
VAC STILL SECTION MATERIAL RECOVERY, W%	73.7	55.2	110.6	107.4	114.2	5.9	119.3	110.0
FEED RATES, KGS/HR								
Feed to Vacuum Still	18.2	64.6	51.4	54.6	49.6	50.9	68.2	35.1
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	13.5	9.4	19.6	21.8	17.1	1.6	23.2	17.4
Vacuum Still Bottoms Product	0.0	26.3	37.3	36.8	39.5	1.5	58.2	21.2

Condition	L/O	L/O	L/O	L/O	L/O	L/O	L/O	L/O
Period	01T	02T	03T	04T	05T	06T	07T	08T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	26.8	59.7	58.8	33.2	47.8	61.0	84.1	78.3
Atmospheric Still Bottoms	4.6	12.3	17.8	23.3	19.3	12.4	17.6	24.0
ROSE Unit DAO	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Vacuum Still Overheads	7.9	0.0	8.4	13.8	10.6	0.0	7.2	5.6
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	68.1	82.9	69.1	47.3	61.5	83.1	77.2	72.5
Atmospheric Still Bottoms	11.8	17.1	20.9	33.1	24.8	16.9	16.2	22.2
ROSE Unit DAO	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Vacuum Still Overheads	20.1	0.0	9.9	19.6	13.7	0.0	6.6	5.2
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	2.89	2.90	4.18	2.75	3.72	0.45	3.54	4.91
C4-C7	1.33	1.26	2.32	1.22	1.83	0.14	1.68	2.61
CO & CO2	0.01	0.01	0.04	0.04	0.04	0.01	0.02	0.21
H2S	1.73	1.63	2.13	1.27	1.62	0.43	1.42	1.46
Net Water	9.2	5.5	9.6	9.4	0.7	-3.5	4.1	5.0
Mix Tank Vent Drain	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unit Knockouts	0.0	0.2	0.2	5.2	0.6	0.0	0.2	0.2
Naphtha Stabilizer Bottoms	56.0	63.4	58.6	61.9	63.9	11.7	52.8	71.8
Atmospheric Still Bottoms	0.0	0.1	2.0	0.1	0.0	0.0	0.1	0.1
Vacuum Still Overhead (Net)	0.1	0.1	0.1	0.1	0.0	0.1	0.1	0.1
Vacuum Still Bottoms (Net)	0.0	26.3	37.3	36.8	39.5	1.5	58.2	21.2
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit Residuals	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL:	83.6	119.8	134.9	136.5	129.5	24.2	150.0	136.1

POC-01 (RUN 260-04) MATERIAL BALANCE

**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)

CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)

Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	L/O	L/O	L/O	L/O	1	1	1	1
Period	09T	10T	11T	12T	13T	14T	15T	16T
Period Start Date	11/10/93	11/11/93	12/04/93	12/05/93	12/06/93	12/07/93	12/08/93	12/09/93
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	12	24	24	24	24	24	24
Solids Separation Type	VAC STIL	VAC STIL	VAC STIL	VAC STIL	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS								

Coal Feed Wet (Less Sample)	1837.1	948.0	1083.5	1761.4	1777.8	1723.4	1376.6	1411.4
Make-Up Oil to Mix Tank	1058.4	294.8	793.7	430.6	317.4	172.0	734.8	668.5
Mix Tank Inventory Loss	1.8	20.9	-43.1	49.0	-176.9	-13.2	50.8	-84.4
Seal Oil to Ebullating Pumps	44.7	21.4	57.9	63.5	63.5	62.1	67.8	67.4
Make-Up Oil to Purge Pumps	111.3	19.5	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	687.0	338.7	617.8	519.9	481.4	541.0	563.6	552.0
Fresh Hydrogen Feed	150.9	74.1	82.8	126.0	129.7	127.7	122.7	112.9
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Make-Up Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	3891.2	1717.5	2592.7	2950.4	2592.8	2613.1	2916.4	2727.9
STREAMS OUT, KGS								

Vent Gas (Dry & N2-Free)	58.6	32.4	33.9	56.5	55.4	40.6	58.6	47.8
Bottoms Flash Gas (Dry & N2-Free)	189.8	74.1	79.2	105.4	130.5	160.1	131.9	198.8
Mix Tank Vent Drain	0.0	0.0	5.2	7.4	0.7	0.3	0.0	0.3
Unit Knockouts	1.5	16.2	3.6	0.0	0.6	0.0	0.0	21.9
Naphtha Stabilizer Bottoms	1744.7	631.4	1201.6	949.1	1115.1	1175.5	1326.0	1043.3
Atmospheric Still Bottoms Product	1.8	0.0	1.5	1.5	1.6	1.2	1.5	1.1
Separated Water (Plus Water in Gases)	743.7	352.3	273.1	729.9	662.8	809.0	541.9	589.0
Vacuum Still Overhead Product	2.1	0.0	1.1	1.4	1.0	1.3	0.2	34.0
Vacuum Still Bottoms Product	812.0	218.6	454.9	1055.5	344.8	0.4	0.6	0.6
ROSE Unit DAO Product	0.0	0.0	0.0	0.0	15.3	16.9	29.3	2.3
ROSE Unit Bottoms	0.0	0.0	0.0	0.0	161.5	478.6	450.9	387.8
ROSE Section Net Inv. Change	0.0	0.0	0.0	0.0	362.4	6.4	-34.0	-24.0
Recycle Oil Net Inv. Change	71.4	32.0	-99.5	-102.3	98.1	-65.3	-79.0	245.4
Vacuum Still Feed Tank Inv. Change	-26.3	168.3	-220.4	-194.1	-222.7	-75.3	76.2	679.5
RLFV Bottoms Holding Tank Inv. Change	-7.4	3.1	-263.3	186.0	-159.0	-17.8	273.7	-193.3
TOTAL PRODUCTS:	3592.1	1528.3	1470.8	2796.3	2568.0	2531.8	2777.8	3034.5
OVERALL UNIT MATERIAL RECOVERY, W%	92.3	89.0	56.7	94.8	99.0	96.9	95.2	111.2

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition	L/O	L/O	L/O	L/O	1	1	1	1
Period	09T	10T	11T	12T	13T	14T	15T	16T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1837.1	948.0	1083.5	1761.4	1777.8	1723.4	1376.6	1411.4
Oil Streams to the SMT								
Recycle to SMT	1885.2	981.6	3020.9	2835.7	2363.6	2343.8	1344.6	2044.4
Make-Up Oil to SMT	1058.4	294.8	793.7	430.6	317.4	172.0	734.8	668.5
VSOH recycled to SMT	0.0	0.0	400.1	338.3	383.9	287.9	268.1	587.1
Mix Tank Inventory Loss	1.8	20.9	-43.1	49.0	-176.9	-13.2	50.8	-84.4
Seal Oil to Ebullating Pumps	44.7	21.4	57.9	63.5	63.5	62.1	67.8	67.4
VSO to Purge Pumps	146.5	117.3	244.7	248.1	219.6	207.7	206.5	201.3
Make Up Oil to Purge Pumps	111.3	19.5	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	687.0	338.7	617.8	519.9	481.4	541.0	563.6	552.0
Fresh Hydrogen Feed	150.9	74.1	82.8	126.0	129.7	127.7	122.7	112.9
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	5922.9	2816.3	6258.3	6372.5	5559.8	5452.5	4735.5	5560.7
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	58.6	32.4	33.9	56.5	55.4	40.6	58.6	47.8
Bottoms Flash Gas (Dry & N2-Free)	189.8	74.1	79.2	105.4	130.5	160.1	131.9	198.8
Mix Tank Vent Drain	0.0	0.0	5.2	7.4	0.7	0.3	0.0	0.3
Unit Knockouts	1.5	16.2	3.6	0.0	0.6	0.0	0.0	21.9
Naphtha Stabilizer Bottoms	1744.7	631.4	1201.6	949.1	1115.1	1175.5	1326.0	1043.3
Atmospheric Still Bottoms	606.4	403.7	190.2	559.9	735.5	717.4	335.7	490.1
Separated Water (Plus Water in Gases)	743.7	352.3	273.1	729.9	662.8	809.0	541.9	589.0
Reactor Liquid Flash Vessel Bottoms	2219.0	1118.6	4496.4	3800.8	2917.0	2444.1	2210.4	3484.3
TOTAL PRODUCTS:	5563.8	2628.6	6283.2	6209.0	5617.6	5347.0	4604.5	5875.5
LIQUEFACTION SECTION RECOVERY, W%	93.9	93.3	100.4	97.4	101.0	98.1	97.2	105.7
SOLVENT TO COAL (MF) RATIO	1.64	1.41	4.07	2.14	1.81	1.70	1.79	2.45

Condition	L/O	L/O	L/O	L/O	1	1	1	1
Period	09T	10T	11T	12T	13T	14T	15T	16T

VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)

STREAMS IN, KGS								
Feed to Vacuum Still	960.7	296.6	3986.1	1685.5				
STREAMS OUT, KGS								
Vacuum Still Overhead Product	148.7	117.3	645.9	587.8	604.4	496.9	474.8	822.5
Vacuum Still Bottoms Product	812.0	218.6	454.9	1055.5	344.8	0.4	0.6	0.6
VAC STILL SECTION MATERIAL RECOVERY, W%	99.9	113.2	27.6	97.5				
FEED RATES, KGS/HR								
Feed to Vacuum Still	40.0	24.7	166.1	70.2				
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	6.2	9.8	26.9	24.5	25.2	20.7	19.8	34.3
Vacuum Still Bottoms Product	33.8	18.2	19.0	44.0	14.4	0.0	0.0	0.0

ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)

STREAMS IN, KGS								
Feed to ROSE Unit					179.6	676.8	706.7	841.9
Makeup Solvent to Rose Unit					0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product					30.3	205.6	250.3	469.1
ROSE Unit Residuals					161.5	478.6	450.9	387.8
ROSE SECTION MATERIAL RECOVERY, W%					106.7	101.1	99.2	101.8
FEED RATES, KGS/HR								
Feed to ROSE Section					7.5	28.2	29.4	35.1
Makeup Solvent to Rose Unit					0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product					1.3	8.6	10.4	19.5
ROSE Unit Residuals					6.7	19.9	18.8	16.2

Condition	L/O	L/O	L/O	L/O	1	1	1	1
Period	09T	10T	11T	12T	13T	14T	15T	16T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	54.3	49.2	117.7	94.0	68.5	58.9	31.4	49.6
Atmospheric Still Bottoms	24.3	32.6	8.1	24.1	29.4	30.7	14.8	18.2
ROSE Unit DAO	0.0	0.0	0.0	0.0	0.6	8.1	9.8	17.4
Vacuum Still Overheads	0.0	0.0	16.7	14.1	16.0	12.0	11.2	24.5
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	69.1	60.2	82.6	71.1	59.9	53.7	46.8	45.3
Atmospheric Still Bottoms	30.9	39.8	5.7	18.3	25.6	28.0	22.0	16.6
ROSE Unit DAO	0.0	0.0	0.0	0.0	0.5	7.4	14.6	15.8
Vacuum Still Overheads	0.0	0.0	11.7	10.7	14.0	10.9	16.6	22.3
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	4.89	4.32	1.23	2.90	3.54	4.37	4.03	5.10
C4-C7	2.63	2.03	0.70	1.00	1.62	1.70	1.47	2.36
CO & CO2	0.04	0.03	0.06	0.05	0.04	0.03	0.03	0.06
H2S	1.52	1.08	1.49	1.27	1.26	1.34	1.15	1.59
Net Water	2.4	1.1	-14.4	8.7	7.6	11.2	-0.9	1.5
Mix Tank Vent Drain	0.0	0.0	0.2	0.3	0.0	0.0	0.0	0.0
Unit Knockouts	0.1	1.3	0.1	0.0	0.0	0.0	0.0	0.9
Naphtha Stabilizer Bottoms	72.7	52.6	50.1	39.5	46.5	-9.0	55.2	43.5
Atmospheric Still Bottoms	0.1	0.0	0.1	0.1	0.1	0.1	0.1	0.0
Vacuum Still Overhead (Net)	0.1	0.0	0.0	0.1	0.0	0.1	0.0	1.4
Vacuum Still Bottoms (Net)	33.8	18.2	19.0	44.0	14.4	0.0	0.0	0.0
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.0	0.0	0.0	0.0	0.6	0.7	1.2	0.1
ROSE Unit Residuals	0.0	0.0	0.0	0.0	6.7	19.9	18.8	16.2
TOTAL:	146.8	109.0	84.3	119.5	102.4	110.9	104.6	95.7

POC-01 (RUN 260-04) MATERIAL BALANCE

**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)

CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)

Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	1	1	1	2	2	2	2	2
Period	17T	18T	19T	20T	21T	22T	23T	24T
Period Start Date	12/10/93	12/11/93	12/12/93	12/13/93	12/14/93	12/15/93	12/16/93	12/17/93
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	24	24	24
Solids Separation Type	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS								

Coal Feed Wet (Less Sample)	1749.8	1741.4	1724.1	1773.1	1781.7	1818.0	1778.3	1786.5
Make-Up Oil to Mix Tank	0.0	227.9	74.0	349.7	146.5	0.0	141.1	57.6
Mix Tank Inventory Loss	-44.9	37.6	-7.3	-29.0	60.3	1.4	-38.1	0.0
Seal Oil to Ebullating Pumps	64.8	70.3	71.8	70.7	68.9	67.2	67.9	68.6
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	552.7	557.8	559.0	557.6	570.9	681.2	669.8	662.6
Fresh Hydrogen Feed	115.3	125.2	123.5	124.9	125.8	122.9	122.7	117.8
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Make-Up Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	2437.6	2760.3	2545.2	2846.9	2754.2	2690.7	2741.7	2693.1
STREAMS OUT, KGS								

Vent Gas (Dry & N2-Free)	47.0	54.4	48.8	45.7	41.9	36.6	36.6	26.1
Bottoms Flash Gas (Dry & N2-Free)	156.0	138.4	129.3	124.6	113.8	131.0	149.3	163.0
Mix Tank Vent Drain	0.1	0.1	0.0	0.4	0.3	0.4	0.8	0.1
Unit Knockouts	20.0	9.9	3.0	14.2	15.6	27.4	45.8	38.8
Naphtha Stabilizer Bottoms	1263.5	1041.8	1081.7	1008.1	922.3	1049.9	1027.7	999.9
Atmospheric Still Bottoms Product	1.6	1.6	2.1	1.6	15.9	1.8	1.7	1.7
Separated Water (Plus Water in Gases)	604.0	776.9	824.4	745.9	675.2	870.2	914.0	931.8
Vacuum Still Overhead Product	413.3	1.3	1.8	1.2	1.2	43.6	2.2	2.1
Vacuum Still Bottoms Product	1.0	1.0	1.5	0.9	148.4	0.9	1.0	0.7
ROSE Unit DAO Product	-30.8	9.5	-15.8	17.0	1.2	5.9	-25.3	28.3
ROSE Unit Bottoms	414.1	500.8	459.1	567.8	331.9	511.4	377.8	404.0
ROSE Section Net Inv. Change	200.9	-89.4	13.6	-70.3	372.4	-141.1	43.1	24.0
Recycle Oil Net Inv. Change	-158.5	76.8	7.7	-11.2	-145.9	-1.8	38.0	-29.0
Vacuum Still Feed Tank Inv. Change	-641.4	66.7	-83.5	285.8	131.1	-97.1	71.7	65.8
RLFV Bottoms Holding Tank Inv. Change	-3.7	1.2	21.5	8.6	-31.3	16.6	-1.2	8.0
TOTAL PRODUCTS:	2287.3	2591.0	2495.1	2740.3	2594.0	2455.9	2683.1	2665.1
OVERALL UNIT MATERIAL RECOVERY, W%	93.8	93.9	98.0	96.3	94.2	91.3	97.9	99.0

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition	1	1	1	2	2	2	2	2
Period	17T	18T	19T	20T	21T	22T	23T	24T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1749.8	1741.4	1724.1	1773.1	1781.7	1818.0	1778.3	1786.5
Oil Streams to the SMT								
Recycle to SMT	1900.4	1522.9	1474.2	1252.4	1151.9	1299.2	1150.8	1261.4
Make-Up Oil to SMT	0.0	227.9	74.0	349.7	146.5	0.0	141.1	57.6
VSOH recycled to SMT	288.5	473.5	605.7	536.2	689.6	878.0	806.9	812.0
Mix Tank Inventory Loss	-44.9	37.6	-7.3	-29.0	60.3	1.4	-38.1	0.0
Seal Oil to Ebullating Pumps	64.8	70.3	71.8	70.7	68.9	67.2	67.9	68.6
VSO to Purge Pumps	200.8	220.0	256.1	253.1	225.4	225.3	227.5	235.0
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	552.7	557.8	559.0	557.6	570.9	681.2	669.8	662.6
Fresh Hydrogen Feed	115.3	125.2	123.5	124.9	125.8	122.9	122.7	117.8
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	4827.2	4976.8	4881.2	4888.5	4821.2	5093.1	4926.9	5001.5
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	47.0	54.4	48.8	45.7	41.9	36.6	36.6	26.1
Bottoms Flash Gas (Dry & N2-Free)	156.0	138.4	129.3	124.6	113.8	131.0	149.3	163.0
Mix Tank Vent Drain	0.1	0.1	0.0	0.4	0.3	0.4	0.8	0.1
Unit Knockouts	20.0	9.9	3.0	14.2	15.6	27.4	45.8	38.8
Naphtha Stabilizer Bottoms	1263.5	1041.8	1081.7	1008.1	922.3	1049.9	1027.7	999.9
Atmospheric Still Bottoms	416.2	464.7	540.9	648.0	856.9	824.2	824.0	860.3
Separated Water (Plus Water in Gases)	604.0	776.9	824.4	745.9	675.2	870.2	914.0	931.8
Reactor Liquid Flash Vessel Bottoms	2133.3	2335.4	2000.5	2124.8	2039.7	1910.1	1871.7	1947.7
TOTAL PRODUCTS:	4640.1	4821.6	4628.6	4711.7	4665.6	4849.8	4869.9	4967.6
LIQUEFACTION SECTION RECOVERY, W%	96.1	96.9	94.8	96.4	96.8	95.2	98.8	99.3
SOLVENT TO COAL (MF) RATIO	1.31	1.34	1.31	1.26	1.17	1.25	1.23	1.24

Condition	1	1	1	2	2	2	2	2
Period	17T	18T	19T	20T	21T	22T	23T	24T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to Vacuum Still								
STREAMS OUT, KGS								
Vacuum Still Overhead Product	902.5	694.9	863.5	790.5	916.2	1146.8	1036.7	1049.0
Vacuum Still Bottoms Product	1.0	1.0	1.5	0.9	148.4	0.9	1.0	0.7
VAC STILL SECTION MATERIAL RECOVERY, W%								
FEED RATES, KGS/HR								
Feed to Vacuum Still								
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	37.6	29.0	36.0	32.9	38.2	47.8	43.2	43.7
Vacuum Still Bottoms Product	0.0	0.0	0.1	0.0	6.2	0.0	0.0	0.0
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to ROSE Unit	836.4	675.2	621.8	759.8	491.1	975.7	726.7	786.5
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	409.3	178.5	173.7	301.5	210.1	461.4	353.8	424.4
ROSE Unit Residuals	414.1	500.8	459.1	567.8	331.9	511.4	377.8	404.0
ROSE SECTION MATERIAL RECOVERY, W%	98.4	100.6	101.8	114.4	110.4	99.7	100.7	105.3
FEED RATES, KGS/HR								
Feed to ROSE Section	34.9	28.1	25.9	31.7	20.5	40.7	30.3	32.8
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	17.1	7.4	7.2	12.6	8.8	19.2	14.7	17.7
ROSE Unit Residuals	17.3	20.9	19.1	23.7	13.8	21.3	15.7	16.8

Condition	1	1	1	2	2	2	2	2
Period	17T	18T	19T	20T	21T	22T	23T	24T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	40.3	38.4	31.2	13.0	0.0	0.0	0.0	0.0
Atmospheric Still Bottoms	18.8	18.4	22.3	27.2	38.4	34.8	32.8	36.0
ROSE Unit DAO	20.0	6.7	7.9	12.0	9.6	19.3	15.1	16.6
Vacuum Still Overheads	12.0	19.7	25.2	22.3	28.7	36.6	33.6	33.8
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	44.2	46.1	36.0	17.5	0.0	0.0	0.0	0.0
Atmospheric Still Bottoms	20.7	22.1	25.8	36.5	50.1	38.4	40.2	41.6
ROSE Unit DAO	21.9	8.1	9.1	16.1	12.4	21.3	18.5	19.2
Vacuum Still Overheads	13.2	23.7	29.1	30.0	37.4	40.3	41.2	39.2
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	4.31	4.08	3.48	3.05	2.84	3.38	3.80	4.24
C4-C7	1.87	1.59	1.61	1.28	1.32	1.53	1.79	1.61
CO & CO2	0.05	0.03	0.04	0.45	0.02	0.02	0.02	0.03
H2S	1.26	1.11	1.16	1.17	1.18	1.14	1.26	1.41
Net Water	2.1	9.1	11.1	7.8	4.3	7.9	10.2	11.2
Mix Tank Vent Drain	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unit Knockouts	0.8	0.4	0.1	0.6	0.6	1.1	1.9	1.6
Naphtha Stabilizer Bottoms	52.6	43.4	45.1	42.0	38.4	43.7	42.8	41.7
Atmospheric Still Bottoms	0.1	0.1	0.1	0.1	0.7	0.1	0.1	0.1
Vacuum Still Overhead (Net)	17.2	0.1	0.1	0.0	0.0	1.8	0.1	0.1
Vacuum Still Bottoms (Net)	0.0	0.0	0.1	0.0	6.2	0.0	0.0	0.0
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	-1.3	0.4	-0.7	0.7	0.1	0.2	-1.1	1.2
ROSE Unit Residuals	17.3	20.9	19.1	23.7	13.8	21.3	15.7	16.8
TOTAL:	119.4	104.4	104.5	103.7	93.3	110.7	104.6	107.6

POC-01 (RUN 260-04) MATERIAL BALANCE
**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	2	2	3A	3A	3A	3A	3A	3A
Period	25T	26T	27T	28T	29T	30T	31T	32T
Period Start Date	12/18/93	12/19/93	12/20/93	12/21/93	12/22/93	12/23/93	12/24/93	12/25/93
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	24	24	24
Solids Separation Type	ROSE-SR	ROSE-SR	VAC STIL	ROSE-SR	VAC STIL	VAC STIL	VAC STIL	VAC STIL
STREAMS IN, KGS								

Coal Feed Wet (Less Sample)	1776.6	1696.0	1860.3	2464.7	2602.0	2661.9	2654.6	2406.2
Make-Up Oil to Mix Tank	44.1	2.3	206.0	155.2	323.3	0.0	466.8	984.7
Mix Tank Inventory Loss	0.0	-0.9	-5.4	5.9	43.5	10.0	10.4	-83.9
Seal Oil to Ebullating Pumps	73.3	74.3	73.8	74.5	74.7	74.9	76.6	78.0
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	95.5
Water Injected to O-1	697.9	648.5	617.7	759.7	795.3	800.0	589.4	507.2
Fresh Hydrogen Feed	115.4	116.7	119.1	130.5	141.2	147.5	156.5	152.6
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Make-Up Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	2707.2	2536.9	2871.5	3590.6	3980.0	3694.3	3954.3	4140.3
STREAMS OUT, KGS								

Vent Gas (Dry & N2-Free)	22.1	21.4	35.5	19.1	25.1	22.2	36.2	39.5
Bottoms Flash Gas (Dry & N2-Free)	123.9	148.3	126.8	168.2	202.3	179.3	189.2	161.5
Mix Tank Vent Drain	0.8	0.0	0.4	0.5	0.2	0.1	0.8	0.7
Unit Knockouts	32.9	41.3	37.4	45.4	23.0	29.6	172.3	241.6
Naptha Stabilizer Bottoms	971.1	988.1	992.9	1246.3	1166.1	733.1	536.0	812.0
Atmospheric Still Bottoms Product	1.6	2.4	1.6	1.8	1.8	1.3	1898.5	1213.8
Separated Water (Plus Water in Gases)	920.4	796.5	819.9	1113.3	1234.1	1313.7	832.3	466.9
Vacuum Still Overhead Product	38.1	16.2	2.2	43.2	143.7	338.1	1.3	0.8
Vacuum Still Bottoms Product	1.0	1.0	849.8	0.9	1605.3	1455.5	664.6	90.7
ROSE Unit DAO Product	-0.9	-3.0	0.0	17.5	0.0	0.0	0.0	0.0
ROSE Unit Bottoms	415.2	424.2	0.2	557.3	0.0	0.0	0.0	0.0
ROSE Section Net Inv. Change	16.3	35.4	0.0	60.8	0.0	0.0	0.0	0.0
Recycle Oil Net Inv. Change	18.0	5.2	16.1	166.1	-157.6	34.4	-204.3	318.4
Vacuum Still Feed Tank Inv. Change	69.9	17.2	29.9	102.5	-199.1	-269.9	-189.1	-115.2
RLFV Bottoms Holding Tank Inv. Change	-4.9	3.1	-12.9	14.7	24.6	-20.3	-73.7	148.5
TOTAL PRODUCTS:	2625.5	2497.2	2899.8	3557.8	4069.5	3817.3	3864.1	3379.1
OVERALL UNIT MATERIAL RECOVERY, W%	97.0	98.4	101.0	99.1	102.2	103.3	97.7	81.6

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition	2	2	3A	3A	3A	3A	3A	3A
Period	25T	26T	27T	28T	29T	30T	31T	32T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1776.6	1696.0	1860.3	2464.7	2602.0	2661.9	2654.6	2406.2
Oil Streams to the SMT								
Recycle to SMT	1280.9	1297.7	1086.8	1760.0	1823.1	2224.7	2346.3	1979.9
Make-Up Oil to SMT	44.1	2.3	206.0	155.2	323.3	0.0	466.8	984.7
VSOH recycled to SMT	796.9	819.6	813.2	1086.5	1061.2	1007.0	367.8	1.4
Mix Tank Inventory Loss	0.0	-0.9	-5.4	5.9	43.5	10.0	10.4	-83.9
Seal Oil to Ebullating Pumps	73.3	74.3	73.8	74.5	74.7	74.9	76.6	78.0
VSO to Purge Pumps	230.7	231.1	259.0	229.4	189.4	215.9	201.4	95.4
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	95.5
Water Injected to O-1	697.9	648.5	617.7	759.7	795.3	800.0	589.4	507.2
Fresh Hydrogen Feed	115.4	116.7	119.1	130.5	141.2	147.5	156.5	152.6
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	5015.7	4885.3	5030.6	6666.4	7053.8	7141.8	6869.8	6217.0
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	22.1	21.4	35.5	19.1	25.1	22.2	36.2	39.5
Bottoms Flash Gas (Dry & N2-Free)	123.9	148.3	126.8	168.2	202.3	179.3	189.2	161.5
Mix Tank Vent Drain	0.8	0.0	0.4	0.5	0.2	0.1	0.8	0.7
Unit Knockouts	32.9	41.3	37.4	45.4	23.0	29.6	172.3	241.6
Naphtha Stabilizer Bottoms	971.1	988.1	992.9	1246.3	1166.1	733.1	536.0	812.0
Atmospheric Still Bottoms	936.4	880.6	940.6	1339.0	1304.5	2021.1	2807.9	3512.1
Separated Water (Plus Water in Gases)	920.4	796.5	819.9	1113.3	1234.1	1313.7	832.3	466.9
Reactor Liquid Flash Vessel Bottoms	1982.5	1938.9	1938.1	2779.4	2939.3	2742.0	2441.4	276.5
TOTAL PRODUCTS:	4990.1	4815.0	4891.5	6711.4	6894.6	7041.2	7016.2	5510.8
LIQUEFACTION SECTION RECOVERY, W%	99.5	98.6	97.2	100.7	97.7	98.6	102.1	88.6
SOLVENT TO COAL (MF) RATIO	1.24	1.30	1.18	1.27	1.29	1.27	1.25	1.29

Condition	2	2	3A	3A	3A	3A	3A	3A
Period	25T	26T	27T	28T	29T	30T	31T	32T

VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)

	25T	26T	27T	28T	29T	30T	31T	32T
STREAMS IN, KGS								
Feed to Vacuum Still			1920.5		3114.8	3031.3	1468.7	192.8
STREAMS OUT, KGS								
Vacuum Still Overhead Product	1065.7	1066.8	1074.4	1359.1	1394.3	1560.9	570.5	97.6
Vacuum Still Bottoms Product	1.0	1.0	849.8	0.9	1605.3	1455.5	664.6	90.7
VAC STILL SECTION MATERIAL RECOVERY, W%			100.2		96.3	99.5	84.1	97.5
FEED RATES, KGS/HR								
Feed to Vacuum Still			80.0		129.8	126.3	61.2	8.0
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	44.4	44.5	44.8	56.6	58.1	65.0	23.8	4.1
Vacuum Still Bottoms Product	0.0	0.0	35.4	0.0	66.9	60.6	27.7	3.8

ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)

	25T	26T	27T	28T	29T	30T	31T	32T
STREAMS IN, KGS								
Feed to ROSE Unit	812.8	860.0	0.0	1222.4	0.0	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	437.0	447.2	0.0	662.0	0.0	0.0	0.0	0.0
ROSE Unit Residuals	415.2	424.2	0.2	557.3	0.0	0.0	0.0	0.0
ROSE SECTION MATERIAL RECOVERY, W%	104.8	101.3		99.7				
FEED RATES, KGS/HR								
Feed to ROSE Section	33.9	35.8	0.0	50.9	0.0	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	18.2	18.6	0.0	27.6	0.0	0.0	0.0	0.0
ROSE Unit Residuals	17.3	17.7	0.0	23.2	0.0	0.0	0.0	0.0

Condition	2	2	3A	3A	3A	3A	3A	3A
Period	25T	26T	27T	28T	29T	30T	31T	32T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	56.3	0.0
Atmospheric Still Bottoms	36.3	35.7	45.3	49.5	76.0	92.7	41.5	82.5
ROSE Unit DAO	17.0	18.3	0.0	23.9	0.0	0.0	0.0	0.0
Vacuum Still Overheads	33.2	34.1	33.9	45.3	44.2	42.0	15.3	0.1
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	49.7	0.0
Atmospheric Still Bottoms	42.0	40.5	57.2	41.7	63.2	68.8	36.7	99.9
ROSE Unit DAO	19.7	20.8	0.0	20.1	0.0	0.0	0.0	0.0
Vacuum Still Overheads	38.4	38.7	42.8	38.2	36.8	31.2	13.6	0.1
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	3.22	3.48	3.41	4.08	4.56	4.79	5.20	3.63
C4-C7	1.23	1.76	1.31	1.34	2.40	1.48	1.64	1.87
CO & CO2	0.03	0.04	0.07	0.11	0.11	0.09	0.11	0.10
H2S	1.01	1.22	1.19	1.66	1.82	1.43	1.67	2.08
Net Water	9.3	6.2	8.4	14.7	18.3	21.4	10.1	-1.7
Mix Tank Vent Drain	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unit Knockouts	1.4	1.7	1.6	1.9	1.0	1.2	7.2	10.1
Naphtha Stabilizer Bottoms	40.5	41.2	41.4	51.9	48.6	30.5	22.3	33.8
Atmospheric Still Bottoms	0.1	0.1	0.1	0.1	0.1	0.1	79.1	50.6
Vacuum Still Overhead (Net)	1.6	0.7	0.1	1.8	6.0	14.1	0.1	0.0
Vacuum Still Bottoms (Net)	0.0	0.0	35.4	0.0	66.9	60.6	27.7	3.8
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.0	-0.1	0.0	0.7	0.0	0.0	0.0	0.0
ROSE Unit Residuals	17.3	17.7	0.0	23.2	0.0	0.0	0.0	0.0
TOTAL:	104.6	100.9	118.6	133.2	182.7	169.0	179.6	125.4

POC-01 (RUN 260-04) MATERIAL BALANCE
**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	L/O	L/O	L/O	L/O	L/O	L/O	L/O	L/O
Period	33T	34T	35T	36T	37T	38T	39T	40T
Period Start Date	01/03/94	01/04/94	01/05/94	01/06/94	01/14/94	01/21/94	01/29/94	01/30/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	12	24	24	24	24
Solids Separation Type	VAC STIL	ROSE-SR	VAC STIL	VAC STIL	VAC STIL	VAC STIL	VAC STIL	VAC STIL
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1446.7	1760.7	1730.3	873.3	2042.8	1500.9	873.5	1708.2
Make-Up Oil to Mix Tank	1218.8	435.5	177.8	345.9	320.1	1234.7	899.1	0.0
Mix Tank Inventory Loss	-76.2	32.7	-6.8	29.0	-97.1	-30.8	-78.0	-10.0
Seal Oil to Ebullating Pumps	59.1	63.4	65.2	34.6	73.0	62.1	54.9	67.9
Make-Up Oil to Purge Pumps	153.4	0.0	116.2	14.7	0.0	131.1	0.0	0.0
Water Injected to O-1	601.2	865.9	1167.6	523.4	1120.4	738.6	658.1	665.1
Fresh Hydrogen Feed	88.5	134.1	112.5	36.2	99.1	50.5	97.1	132.8
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Make-Up Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	3491.5	3292.1	3362.8	1857.1	3558.2	3687.1	2504.7	2564.0
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	32.3	40.4	32.4	40.3	25.0	15.1	30.8	41.4
Bottoms Flash Gas (Dry & N2-Free)	58.4	89.9	109.9	40.9	119.7	32.1	17.2	106.8
Mix Tank Vent Drain	18.8	15.3	2.4	1.4	2.0	4.7	2.6	0.4
Unit Knockouts	25.5	174.1	55.6	32.0	85.3	55.4	78.6	97.0
Naphtha Stabilizer Bottoms	595.8	1248.9	1142.1	577.9	857.2	792.0	717.0	883.0
Atmospheric Still Bottoms Product	1.3	1.6	1.5	0.0	1.6	0.0	1.2	1.1
Separated Water (Plus Water in Gases)	743.0	1245.5	1128.7	635.1	1526.2	914.1	649.3	838.5
Vacuum Still Overhead Product	1.8	128.2	1.1	0.0	1.8	0.0	296.8	455.2
Vacuum Still Bottoms Product	726.0	0.9	700.8	246.8	913.0	234.5	4.0	232.2
ROSE Unit DAO Product	0.0	34.1	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit Bottoms	0.0	152.7	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Section Net Inv. Change	0.0	376.0	0.0	0.0	0.0	0.0	0.0	0.0
Recycle Oil Net Inv. Change	-10.1	-7.4	-238.4	72.0	150.4	5.3	20.2	534.9
Vacuum Still Feed Tank Inv. Change	136.5	-406.4	-130.2	70.8	-236.8	287.6	546.1	-512.1
RLFV Bottoms Holding Tank Inv. Change	-24.6	6.8	-104.3	33.1	-214.8	16.6	-32.5	31.3
TOTAL PRODUCTS:	2304.8	3100.6	2701.7	1750.2	3230.7	2357.3	2331.3	2709.6
OVERALL UNIT MATERIAL RECOVERY, W%	66.0	94.2	80.3	94.2	90.8	63.9	93.1	105.7

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition	L/O	L/O	L/O	L/O	L/O	L/O	L/O	L/O
Period	33T	34T	35T	36T	37T	38T	39T	40T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1446.7	1760.7	1730.3	873.3	2042.8	1500.9	873.5	1708.2
Oil Streams to the SMT								
Recycle to SMT	2687.5	2480.9	2180.0	750.2	3081.3	2908.4	2703.1	2280.2
Make-Up Oil to SMT	1218.8	435.5	177.8	345.9	320.1	1234.7	899.1	0.0
VSOH recycled to SMT	0.0	559.1	178.8	0.0	1064.8	0.0	63.0	108.9
Mix Tank Inventory Loss	-76.2	32.7	-6.8	29.0	-97.1	-30.8	-78.0	-10.0
Seal Oil to Ebullating Pumps	59.1	63.4	65.2	34.6	73.0	62.1	54.9	67.9
VSO to Purge Pumps	77.8	284.4	113.4	98.4	238.8	100.7	248.6	218.1
Make Up Oil to Purge Pumps	153.4	0.0	116.2	14.7	0.0	131.1	0.0	0.0
Water Injected to O-1	601.2	865.9	1167.6	523.4	1120.4	738.6	658.1	665.1
Fresh Hydrogen Feed	88.5	134.1	112.5	36.2	99.1	50.5	97.1	132.8
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	6256.8	6616.5	5834.8	2705.8	7943.1	6696.1	5519.5	5171.3
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	32.3	40.4	32.4	40.3	25.0	15.1	30.8	41.4
Bottoms Flash Gas (Dry & N2-Free)	58.4	89.9	109.9	40.9	119.7	32.1	17.2	106.8
Mix Tank Vent Drain	18.8	15.3	2.4	1.4	2.0	4.7	2.6	0.4
Unit Knockouts	25.5	174.1	55.6	32.0	85.3	55.4	78.6	97.0
Naphtha Stabilizer Bottoms	595.8	1248.9	1142.1	577.9	857.2	792.0	717.0	883.0
Atmospheric Still Bottoms	786.0	830.3	989.4	396.0	718.3	714.9	404.0	731.0
Separated Water (Plus Water in Gases)	743.0	1245.5	1128.7	635.1	1526.2	914.1	649.3	838.5
Reactor Liquid Flash Vessel Bottoms	3908.1	2794.6	1852.1	919.9	5706.2	3928.3	4190.7	2155.1
TOTAL PRODUCTS:	6168.0	6439.2	5312.7	2643.4	9039.9	6456.6	6090.3	4853.1
LIQUEFACTION SECTION RECOVERY, W%	98.6	97.3	91.1	97.7	113.8	96.4	110.3	93.8
SOLVENT TO COAL (MF) RATIO	2.83	2.06	1.53	1.31	2.28	2.88	4.37	1.46

Condition	L/O	L/O	L/O	L/O	L/O	L/O	L/O	L/O
Period	33T	34T	35T	36T	37T	38T	39T	40T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to Vacuum Still	1752.7		1129.0	391.0	2171.3	1426.1	1355.3	1164.8
STREAMS OUT, KGS								
Vacuum Still Overhead Product	79.6	971.6	293.2	98.4	1305.4	100.7	608.4	782.2
Vacuum Still Bottoms Product	726.0	0.9	700.8	246.8	913.0	234.5	4.0	232.2
VAC STILL SECTION MATERIAL RECOVERY, W%	45.9		88.0	88.3	102.1	23.5	45.1	87.0
FEED RATES, KGS/HR								
Feed to Vacuum Still	73.0		47.0	32.6	90.5	59.4	56.5	48.5
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	3.3	40.5	12.2	8.2	54.4	4.2	25.4	32.6
Vacuum Still Bottoms Product	30.2	0.0	29.2	20.6	38.0	9.8	0.2	9.7
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to ROSE Unit	0.0	631.9	0.0	0.0	0.0	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	0.0	435.8	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit Residuals	0.0	152.7	0.0	0.0	0.0	0.0	0.0	0.0
ROSE SECTION MATERIAL RECOVERY, W%		93.1						
FEED RATES, KGS/HR								
Feed to ROSE Section	0.0	26.3	0.0	0.0	0.0	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	0.0	18.2	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit Residuals	0.0	6.4	0.0	0.0	0.0	0.0	0.0	0.0

Condition	L/O	L/O	L/O	L/O	L/O	L/O	L/O	L/O
Period	33T	34T	35T	36T	37T	38T	39T	40T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	79.2	52.0	44.6	32.4	99.9	91.5	96.0	70.4
Atmospheric Still Bottoms	32.8	34.6	46.2	30.1	28.5	29.7	16.7	24.6
ROSE Unit DAO	0.0	16.8	0.0	0.0	0.0	0.0	0.0	0.0
Vacuum Still Overheads	0.0	23.3	7.4	0.0	44.4	0.0	2.6	4.5
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	70.7	41.0	45.4	51.8	57.8	75.5	83.3	70.7
Atmospheric Still Bottoms	29.3	27.3	47.0	48.2	16.5	24.5	14.5	24.7
ROSE Unit DAO	0.0	13.3	0.0	0.0	0.0	0.0	0.0	0.0
Vacuum Still Overheads	0.0	18.4	7.6	0.0	25.7	0.0	2.3	4.6
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	1.25	2.59	3.02	3.50	2.74	0.53	0.49	2.79
C4-C7	0.40	0.94	1.20	1.68	0.96	0.22	0.25	1.04
CO & CO2	0.08	0.06	0.05	0.05	0.11	0.03	0.05	0.07
H2S	1.12	0.76	0.77	1.07	1.71	0.46	0.09	1.10
Net Water	5.9	15.8	-1.6	9.3	16.9	7.3	-0.4	7.2
Mix Tank Vent Drain	0.8	0.6	0.1	0.1	0.1	0.2	0.1	0.0
Unit Knockouts	1.1	7.3	2.3	2.7	3.6	2.3	3.3	4.0
Naphtha Stabilizer Bottoms	24.8	52.0	47.6	48.2	35.7	33.0	29.9	36.8
Atmospheric Still Bottoms	0.1	0.1	0.1	0.0	0.1	0.0	0.0	0.0
Vacuum Still Overhead (Net)	0.1	5.3	0.0	0.0	0.1	0.0	12.4	19.0
Vacuum Still Bottoms (Net)	30.2	0.0	29.2	20.6	38.0	9.8	0.2	9.7
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.0	1.4	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit Residuals	0.0	6.4	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL:	90.8	129.3	131.3	130.7	146.5	84.6	73.7	109.4

POC-01 (RUN 260-04) MATERIAL BALANCE

**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	3B	3B	3B	3B	L/O	L/O	4A/B	4A/B
Period	41T	42T	43T	44T	45T	46T	47T	48T
Period Start Date	01/31/94	02/01/94	02/02/94	02/03/94	02/05/94	02/06/94	02/07/94	02/08/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	24	24	24
Solids Separation Type	VAC STIL	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS								

Coal Feed Wet (Less Sample)	2093.6	2198.4	2197.1	1170.3	1562.0	1704.7	2177.8	2238.4
Make-Up Oil to Mix Tank	691.0	700.8	172.3	933.9	0.0	30.9	625.6	298.8
Mix Tank Inventory Loss	-36.3	-53.1	16.8	-26.3	-71.2	-45.4	6.4	-11.3
Seal Oil to Ebullating Pumps	66.3	58.2	57.4	56.6	54.8	53.9	53.6	54.5
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	669.0	649.0	680.9	510.9	613.8	570.4	604.9	625.5
Fresh Hydrogen Feed	171.7	177.3	174.9	135.0	119.7	153.8	177.5	177.3
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Make-Up Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	3655.3	3730.7	3299.4	2780.4	2279.0	2468.4	3645.8	3383.3
STREAMS OUT, KGS								

Vent Gas (Dry & N2-Free)	43.6	45.9	46.8	50.4	35.2	41.9	46.5	50.5
Bottoms Flash Gas (Dry & N2-Free)	137.3	129.1	139.5	137.3	85.3	139.4	256.0	211.5
Mix Tank Vent Drain	0.3	0.3	0.6	0.0	4.2	1.1	0.4	0.2
Unit Knockouts	43.8	8.6	49.5	222.7	210.7	29.8	23.7	159.5
Naphtha Stabilizer Bottoms	1125.1	1202.7	1179.5	682.1	377.3	955.7	1166.0	1105.8
Atmospheric Still Bottoms Product	1.5	1.3	1.3	0.0	1.7	1.2	1.6	5.0
Separated Water (Plus Water in Gases)	1076.9	995.8	945.1	797.1	817.2	859.8	921.4	919.6
Vacuum Still Overhead Product	9.9	10.7	27.3	4.5	468.6	128.5	2.3	2.3
Vacuum Still Bottoms Product	580.7	0.9	0.7	0.0	0.3	1.0	1.1	0.8
Pressure Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO Product	0.0	44.5	-4.9	0.7	17.8	35.6	-57.3	-14.1
ROSE Unit Bottoms	0.0	329.3	399.6	659.1	518.5	403.7	656.8	703.1
ROSE Section Net Inv. Change	0.0	630.4	96.2	131.2	46.7	-272.2	164.2	-69.4
Recycle Oil Net Inv. Change	-189.5	121.3	-22.2	146.0	248.6	-409.9	65.0	-40.3
Vacuum Still Feed Tank Inv. Change	444.1	28.1	-8.6	-179.6	-280.8	10.0	135.6	412.8
RLFV Bottoms Holding Tank Inv. Change	159.0	-31.3	54.6	-178.6	-40.5	-15.3	27.0	0.6
TOTAL PRODUCTS:	3432.6	3517.5	2904.9	2472.8	2510.9	1910.3	3410.3	3447.9
OVERALL UNIT MATERIAL RECOVERY, W%	93.9	94.3	88.0	88.9	110.2	77.4	93.5	101.9

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition	3B	3B	3B	3B	L/O	L/O	4A/B	4A/B
Period	41T	42T	43T	44T	45T	46T	47T	48T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	2093.6	2198.4	2197.1	1170.3	1562.0	1704.7	2177.8	2238.4
Oil Streams to the SMT								
Recycle to SMT	1478.7	1379.9	1691.7	959.4	2823.8	2270.7	1493.0	1432.2
Make-Up Oil to SMT	691.0	700.8	172.3	933.9	0.0	30.9	625.6	298.8
VSOH recycled to SMT	394.0	815.1	741.5	832.3	185.7	73.4	511.7	669.9
Mix Tank Inventory Loss	-36.3	-53.1	16.8	-26.3	-71.2	-45.4	6.4	-11.3
Seal Oil to Ebullating Pumps	66.3	58.2	57.4	56.6	54.8	53.9	53.6	54.5
VSO to Purge Pumps	209.8	220.2	229.6	204.9	230.3	217.9	220.6	243.4
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	669.0	649.0	680.9	510.9	613.8	570.4	604.9	625.5
Fresh Hydrogen Feed	171.7	177.3	174.9	135.0	119.7	153.8	177.5	177.3
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	5737.8	6145.9	5962.2	4777.0	5518.8	5030.4	5871.1	5728.7
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	43.6	45.9	46.8	50.4	35.2	41.9	46.5	50.5
Bottoms Flash Gas (Dry & N2-Free)	137.3	129.1	139.5	137.3	85.3	139.4	256.0	211.5
Mix Tank Vent Drain	0.3	0.3	0.6	0.0	4.2	1.1	0.4	0.2
Unit Knockouts	43.8	8.6	49.5	222.7	210.7	29.8	23.7	159.5
Naphtha Stabilizer Bottoms	1125.1	1202.7	1179.5	682.1	377.3	955.7	1166.0	1105.8
Atmospheric Still Bottoms	956.8	1310.0	1390.3	716.1	620.5	642.8	897.3	1124.1
Separated Water (Plus Water in Gases)	1076.9	995.8	945.1	797.1	817.2	859.8	921.4	919.6
Reactor Liquid Flash Vessel Bottoms	2196.2	2202.5	2086.8	1793.8	3810.2	1878.0	2577.6	2147.9
TOTAL PRODUCTS:	5580.0	5894.8	5838.1	4399.6	5960.8	4548.5	5888.9	5719.1
LIQUEFACTION SECTION RECOVERY, W%	97.2	95.9	97.9	92.1	108.0	90.4	100.3	99.8
SOLVENT TO COAL (MF) RATIO	1.28	1.37	1.24	2.43	2.01	1.45	1.26	1.12

Condition	3B	3B	3B	3B	L/O	L/O	4A/B	4A/B
Period	41T	42T	43T	44T	45T	46T	47T	48T

VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)

STREAMS IN, KGS								
Feed to Vacuum Still	1265.5							
STREAMS OUT, KGS								
Vacuum Still Overhead Product	613.8	1045.9	998.4	1041.7	884.6	419.8	734.6	915.6
Vacuum Still Bottoms Product	580.7	0.9	0.7	0.0	0.3	1.0	1.1	0.8
VAC STILL SECTION MATERIAL RECOVERY, W%	93.7							
FEED RATES, KGS/HR								
Feed to Vacuum Still	52.7							
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	25.6	43.6	41.6	43.4	36.9	17.5	30.6	38.1
Vacuum Still Bottoms Product	24.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0

ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)

STREAMS IN, KGS								
Feed to ROSE Unit	0.0	411.0	842.8	927.6	848.7	714.4	959.8	1026.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	0.0	198.6	353.0	361.8	449.7	290.8	367.7	361.5
ROSE Unit Residuals	0.0	329.3	399.6	659.1	518.5	403.7	656.8	703.1
ROSE SECTION MATERIAL RECOVERY, W%		128.5	89.3	110.1	114.1	97.2	106.7	103.8
FEED RATES, KGS/HR								
Feed to ROSE Section	0.0	17.1	35.1	38.6	35.4	29.8	40.0	42.8
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	0.0	8.3	14.7	15.1	18.7	12.1	15.3	15.1
ROSE Unit Residuals	0.0	13.7	16.7	27.5	21.6	16.8	27.4	29.3

Condition	3B	3B	3B	3B	L/O	L/O	4A/B	4A/B
Period	41T	42T	43T	44T	45T	46T	47T	48T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	16.0	0.0	0.0	0.0	77.4	49.0	9.5	0.0
Atmospheric Still Bottoms	45.7	51.4	56.0	26.6	23.7	32.6	35.8	44.7
ROSE Unit DAO	0.0	6.1	14.4	13.4	16.5	13.0	17.0	15.0
Vacuum Still Overheads	16.4	34.0	30.9	34.7	7.7	3.1	21.3	27.9
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	20.5	0.0	0.0	0.0	61.7	50.2	11.3	0.0
Atmospheric Still Bottoms	58.5	56.2	55.3	35.6	18.9	33.4	42.8	51.0
ROSE Unit DAO	0.0	6.6	14.2	17.9	13.2	13.3	20.3	17.1
Vacuum Still Overheads	21.0	37.1	30.5	46.5	6.2	3.1	25.5	31.9
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	3.54	3.17	3.96	3.98	2.31	3.97	6.00	5.27
C4-C7	1.45	1.49	1.62	1.60	0.61	1.45	2.82	2.47
CO & CO2	0.40	0.19	0.12	0.13	0.08	0.06	0.13	0.10
H2S	1.11	1.26	0.98	0.97	1.00	1.05	2.56	1.97
Net Water	17.0	14.4	11.0	11.9	8.5	12.1	13.2	12.3
Mix Tank Vent Drain	0.0	0.0	0.0	0.0	0.2	0.0	0.0	0.0
Unit Knockouts	1.8	0.4	2.1	9.3	8.8	1.2	1.0	6.6
Naphtha Stabilizer Bottoms	46.9	50.1	49.1	28.4	15.7	39.8	48.6	46.1
Atmospheric Still Bottoms	0.1	0.1	0.1	0.0	0.1	0.0	0.1	0.2
Vacuum Still Overhead (Net)	0.4	0.4	1.1	0.2	19.5	5.4	0.1	0.1
Vacuum Still Bottoms (Net)	24.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.0	1.9	-0.2	0.0	0.7	1.5	-2.4	-0.6
ROSE Unit Residuals	0.0	13.7	16.7	27.5	21.6	16.8	27.4	29.3
TOTAL:	124.4	114.0	114.8	105.1	104.6	107.2	124.5	129.8

POC-01 (RUN 260-04) MATERIAL BALANCE
**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	4A/B	4A/B	L/O	L/O	L/O	4C	4C	4C
Period	49T	50T	51T	52T	53T	54T	55T	56T
Period Start Date	02/09/94	02/10/94	02/11/94	02/12/94	02/13/94	02/14/94	02/15/94	02/16/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	24	24	24
Solids Separation Type	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	2317.2	2430.5	8.7	682.5	1719.6	1925.9	2524.0	2585.0
Make-Up Oil to Mix Tank	325.1	0.0	2636.7	977.1	0.0	371.8	398.5	88.5
Mix Tank Inventory Loss	21.3	82.6	-203.2	72.1	7.7	-51.3	67.1	21.8
Seal Oil to Ebullating Pumps	54.6	55.5	54.7	55.5	58.2	57.7	58.9	60.3
Make-Up Oil to Purge Pumps	0.0	0.0	99.6	185.0	0.0	0.0	0.0	0.0
Water Injected to O-1	752.0	801.5	466.2	423.8	656.5	686.7	684.3	700.8
Fresh Hydrogen Feed	177.5	186.9	187.5	184.4	183.2	184.1	135.5	178.1
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	3647.8	3557.0	3250.2	2580.4	2625.1	3175.0	3868.3	3634.3
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	54.4	59.5	53.1	43.3	37.3	46.3	41.3	37.5
Bottoms Flash Gas (Dry & N2-Free)	221.2	217.6	93.2	97.6	170.0	226.1	273.8	295.3
Mix Tank Vent Drain	0.0	0.0	14.8	7.2	10.7	1.1	1.0	0.4
Unit Knockouts	34.9	42.2	34.7	32.2	31.4	27.8	27.5	60.5
Naphtha Stabilizer Bottoms	1126.1	1119.8	319.3	333.8	720.2	1126.1	1104.9	1058.4
Atmospheric Still Bottoms Product	1.7	1.8	1.8	0.0	1.5	1.8	1.5	1.9
Separated Water (Plus Water in Gases)	1022.9	1076.2	589.3	530.1	852.7	956.7	1060.7	1141.2
Vacuum Still Overhead Product	2.3	121.5	2.0	0.0	489.7	2.5	2.2	16.0
Vacuum Still Bottoms Product	1.0	1.0	0.8	0.0	1.0	1.1	1.0	2.0
ROSE Unit DAO Product	9.8	2.7	5.1	64.4	12.6	-6.0	17.4	2.6
ROSE Unit Bottoms	525.0	838.5	719.8	5.0	145.1	353.8	662.2	945.3
ROSE Section Net Inv. Change	179.6	88.0	-294.8	-119.3	426.4	205.0	72.6	109.8
Recycle Oil Net Inv. Change	66.8	-18.5	-266.2	453.2	-116.0	-129.0	130.6	60.4
Vacuum Still Feed Tank Inv. Change	174.2	-121.1	-612.3	774.7	-478.5	205.0	238.1	-18.1
RLFV Bottoms Holding Tank Inv. Change	45.4	-19.6	548.7	221.0	-151.0	22.1	187.2	-93.3
TOTAL PRODUCTS:	3465.3	3409.6	1209.3	2443.2	2153.0	3040.3	3822.0	3619.9
OVERALL UNIT MATERIAL RECOVERY, W%	95.0	95.9	37.2	94.7	82.0	95.8	98.8	99.6

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition	4A/B	4A/B	L/O	L/O	L/O	4C	4C	4C
Period	49T	50T	51T	52T	53T	54T	55T	56T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	2317.2	2430.5	8.7	682.5	1719.6	1925.9	2524.0	2585.0
Oil Streams to the SMT								
Recycle to SMT	1517.4	1991.8	1017.6	405.8	2230.8	1416.2	1259.3	1590.4
Make-Up Oil to SMT	325.1	0.0	2636.7	977.1	0.0	371.8	398.5	88.5
VSOH recycled to SMT	693.5	742.3	411.0	0.0	188.7	462.8	687.2	821.1
Mix Tank Inventory Loss	21.3	82.6	-203.2	72.1	7.7	-51.3	67.1	21.8
Seal Oil to Ebullating Pumps	54.6	55.5	54.7	55.5	58.2	57.7	58.9	60.3
VSO to Purge Pumps	243.0	250.5	162.3	86.6	263.6	250.3	242.2	243.2
Make Up Oil to Purge Pumps	0.0	0.0	99.6	185.0	0.0	0.0	0.0	0.0
Water Injected to 0-1	752.0	801.5	466.2	423.8	656.5	686.7	684.3	700.8
Fresh Hydrogen Feed	177.5	186.9	187.5	184.4	183.2	184.1	135.5	178.1
DMDS (TNPS)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL FEED:	6101.8	6541.6	4841.1	3072.9	5308.2	5304.2	6057.1	6289.0
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	54.4	59.5	53.1	43.3	37.3	46.3	41.3	37.5
Bottoms Flash Gas (Dry & N2-Free)	221.2	217.6	93.2	97.6	170.0	226.1	273.8	295.3
Mix Tank Vent Drain	0.0	0.0	14.8	7.2	10.7	1.1	1.0	0.4
Unit Knockouts	34.9	42.2	34.7	32.2	31.4	27.8	27.5	60.5
Naphtha Stabilizer Bottoms	1126.1	1119.8	319.3	333.8	720.2	1126.1	1104.9	1058.4
Atmospheric Still Bottoms	1167.9	1482.5	620.2	228.4	540.4	625.0	1057.9	1230.1
Separated Water (Plus Water in Gases)	1022.9	1076.2	589.3	530.1	852.7	956.7	1060.7	1141.2
Reactor Liquid Flash Vessel Bottoms	2329.7	2605.6	1701.1	1874.8	2549.0	2227.5	2469.1	2455.5
TOTAL PRODUCTS:	5957.1	6603.5	3425.5	3147.4	4911.7	5236.6	6036.2	6278.9
LIQUEFACTION SECTION RECOVERY, W%	97.6	100.9	70.8	102.4	92.5	98.7	99.7	99.8
SOLVENT TO COAL (MF) RATIO	1.14	1.17	488.16	2.11	1.47	1.22	0.97	1.01

Condition	4A/B	4A/B	L/O	L/O	L/O	4C	4C	4C
Period	49T	50T	51T	52T	53T	54T	55T	56T

VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)

STREAMS IN, KGS								
Feed to Vacuum Still								
STREAMS OUT, KGS								
Vacuum Still Overhead Product	938.8	1114.3	575.3	86.6	941.9	715.5	931.6	1080.4
Vacuum Still Bottoms Product	1.0	1.0	0.8	0.0	1.0	1.1	1.0	2.0
VAC STILL SECTION MATERIAL RECOVERY, W%								
FEED RATES, KGS/HR								
Feed to Vacuum Still								
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	39.1	46.4	24.0	3.6	39.2	29.8	38.8	45.0
Vacuum Still Bottoms Product	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1

ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)

STREAMS IN, KGS								
Feed to ROSE Unit	894.9	1400.2	1422.9	119.3	422.3	565.6	1022.4	1367.6
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	386.9	618.4	663.0	95.7	276.5	218.4	379.6	479.2
ROSE Unit Residuals	525.0	838.5	719.8	5.0	145.1	353.8	662.2	945.3
ROSE SECTION MATERIAL RECOVERY, W%	101.9	104.0	97.2	84.4	99.8	101.2	101.9	104.2
FEED RATES, KGS/HR								
Feed to ROSE Section	37.3	58.3	59.3	5.0	17.6	23.6	42.6	57.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	16.1	25.8	27.6	4.0	11.5	9.1	15.8	20.0
ROSE Unit Residuals	21.9	34.9	30.0	0.2	6.0	14.7	27.6	39.4

Condition	4A/B	4A/B	L/O	L/O	L/O	4C	4C	4C
Period	49T	50T	51T	52T	53T	54T	55T	56T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	0.0	0.0	0.0	11.8	57.7	20.2	0.0	0.0
Atmospheric Still Bottoms	47.8	58.6	20.5	4.5	23.7	28.6	39.1	47.7
ROSE Unit DAO	15.4	24.4	21.9	0.6	11.6	10.3	13.4	18.5
Vacuum Still Overheads	28.9	30.9	17.1	0.0	7.9	19.3	28.6	34.2
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	0.0	0.0	0.0	69.8	57.2	25.7	0.0	0.0
Atmospheric Still Bottoms	51.9	51.5	34.5	26.6	23.5	36.5	48.2	47.5
ROSE Unit DAO	16.8	21.4	36.7	3.6	11.5	13.1	16.5	18.4
Vacuum Still Overheads	31.4	27.2	28.8	0.0	7.8	24.6	35.3	34.1
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	5.26	5.75	3.02	2.91	4.26	5.50	6.30	7.14
C4-C7	2.45	2.16	0.94	0.93	1.62	2.22	2.60	2.46
CO & CO2	0.12	0.14	0.08	0.06	0.09	0.18	0.24	0.24
H2S	2.56	2.30	1.04	1.10	1.85	2.52	3.07	3.24
Net Water	11.3	11.4	5.1	4.4	8.2	11.2	15.7	18.4
Mix Tank Vent Drain	0.0	0.0	0.6	0.3	0.4	0.0	0.0	0.0
Unit Knockouts	1.5	1.8	1.4	1.3	1.3	1.2	1.1	2.5
Naphtha Stabilizer Bottoms	46.9	46.7	13.3	13.9	30.0	46.9	46.0	44.1
Atmospheric Still Bottoms	0.1	0.1	0.1	0.0	0.1	0.1	0.1	0.1
Vacuum Still Overhead (Net)	0.1	5.1	0.1	0.0	20.4	0.1	0.1	0.7
Vacuum Still Bottoms (Net)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.4	0.1	0.2	2.7	0.5	-0.3	0.7	0.1
ROSE Unit Residuals	21.9	34.9	30.0	0.2	6.0	14.7	27.6	39.4
TOTAL:	123.8	143.7	75.3	45.5	102.1	112.9	131.9	147.4

POC-01 (RUN 260-04) MATERIAL BALANCE
**** CTSL PDU DATA ****

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Condition	4C	5
Period	57T	58T
Period Start Date	02/17/94	02/18/94
Period Start Time	04:00	04:00
Period Duration Hours	24	24
Solids Separation Type	ROSE-SR	VAC STIL
STREAMS IN, KGS		
Coal Feed Wet (Less Sample)	2597.2	2526.0
Make-Up Oil to Mix Tank	43.3	1059.4
Mix Tank Inventory Loss	-60.3	61.2
Seal Oil to Ebullating Pumps	62.2	63.8
Make-Up Oil to Purge Pumps	0.0	116.9
Water Injected to O-1	586.8	577.1
Fresh Hydrogen Feed	187.2	196.3
DMDS (TNPS)	0.0	0.0
Make-Up Solvent to Rose Unit	0.0	0.0
TOTAL FEED:	3416.4	4600.7
STREAMS OUT, KGS		
Vent Gas (Dry & N2-Free)	45.2	45.1
Bottoms Flash Gas (Dry & N2-Free)	315.9	332.0
Mix Tank Vent Drain	0.5	0.9
Unit Knockouts	21.8	84.8
Naphtha Stabilizer Bottoms	1020.7	1369.2
Atmospheric Still Bottoms Product	0.0	18.3
Separated Water (Plus Water in Gases)	980.3	972.9
Vacuum Still Overhead Product	0.0	13.9
Vacuum Still Bottoms Product	0.0	235.6
Pressure Filter Cake	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0
ROSE Unit DAO Product	-3.1	0.0
ROSE Unit Bottoms	1047.8	0.0
ROSE Section Net Inv. Change	-24.9	0.0
Recycle Oil Net Inv. Change	-154.1	-109.7
Vacuum Still Feed Tank Inv. Change	-244.9	-453.1
RLFV Bottoms Holding Tank Inv. Change	-103.7	2123.0
TOTAL PRODUCTS:	2901.6	4632.9
OVERALL UNIT MATERIAL RECOVERY, W%	84.9	100.7

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Condition Period	4C 57T	5 58T
STREAMS IN, KGS		
Coal Feed Wet (Less Sample)	2597.2	2526.0
Oil Streams to the SMT		
Recycle to SMT	1792.7	1491.4
Make-Up Oil to SMT	43.3	1059.4
VSOH recycled to SMT	833.2	104.7
Mix Tank Inventory Loss	-60.3	61.2
Seal Oil to Ebullating Pumps	62.2	63.8
VSO to Purge Pumps	255.1	125.3
Make Up Oil to Purge Pumps	0.0	116.9
Water Injected to O-1	586.8	577.1
Fresh Hydrogen Feed	187.2	196.3
DMDS (TNPS)	0.0	0.0
TOTAL FEED:	6297.4	6322.2
STREAMS OUT, KGS		
Vent Gas (Dry & N2-Free)	45.2	45.1
Bottoms Flash Gas (Dry & N2-Free)	315.9	332.0
Mix Tank Vent Drain	0.5	0.9
Unit Knockouts	21.8	84.8
Naphtha Stabilizer Bottoms	1020.7	1369.2
Atmospheric Still Bottoms	1476.7	1120.8
Separated Water (Plus Water in Gases)	980.3	972.9
Reactor Liquid Flash Vessel Bottoms	2376.0	2039.7
TOTAL PRODUCTS:	6237.3	5965.4
LIQUEFACTION SECTION RECOVERY, W%	99.0	94.4
SOLVENT TO COAL (MF) RATIO	1.07	1.10

Condition	4C	5
Period	57T	58T

VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)

	4C	5
STREAMS IN, KGS		
Feed to Vacuum Still		453.6
STREAMS OUT, KGS		
Vacuum Still Overhead Product	1088.3	243.8
Vacuum Still Bottoms Product	0.0	235.6
VAC STILL SECTION MATERIAL RECOVERY, W%		105.6
FEED RATES, KGS/HR		
Feed to Vacuum Still		18.9
PRODUCT RATES, KGS/HR		
Vacuum Still Overhead Product	45.3	10.2
Vacuum Still Bottoms Product	0.0	9.8

ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)

	4C	5
STREAMS IN, KGS		
Feed to ROSE Unit	1577.1	0.0
Makeup Solvent to Rose Unit	0.0	0.0
STREAMS OUT, KGS		
ROSE Unit DAO Product	544.9	0.0
ROSE Unit Residuals	1047.8	0.0
ROSE SECTION MATERIAL RECOVERY, W%	101.0	
FEED RATES, KGS/HR		
Feed to ROSE Section	65.7	0.0
Makeup Solvent to Rose Unit	0.0	0.0
PRODUCT RATES, KGS/HR		
ROSE Unit DAO Product	22.7	0.0
ROSE Unit Residuals	43.7	0.0

Condition	4C	5
Period	57T	58T

RECYCLE RATES TO SMT, KGS/HR

Reactor Liq Flash Vessel Bottoms	0.0	0.0
Atmospheric Still Bottoms	54.5	62.1
ROSE Unit DAO	20.2	0.0
Vacuum Still Overheads	34.7	4.4
Vacuum Still Bottoms	0.0	0.0

COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%

Reactor Liq Flash Vessel Bottoms	0.0	0.0
Atmospheric Still Bottoms	49.8	93.4
ROSE Unit DAO	18.5	0.0
Vacuum Still Overheads	31.7	6.6
Vacuum Still Bottoms	0.0	0.0

NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)

GASES: C1-C3	6.66	6.94
C4-C7	3.27	3.43
CO & CO2	1.35	1.41
H2S	2.85	2.99
Net Water	16.4	16.5
Mix Tank Vent Drain	0.0	0.0
Unit Knockouts	0.9	3.5
Naphtha Stabilizer Bottoms	42.5	57.1
Atmospheric Still Bottoms	0.0	0.8
Vacuum Still Overhead (Net)	0.0	0.6
Vacuum Still Bottoms (Net)	0.0	9.8
Filter Cake	0.0	0.0
ROSE Unit DAO	-0.1	0.0
ROSE Unit Residuals	43.7	0.0
TOTAL:	140.6	125.7

APPENDIX D

Inspection of Product & Internal Streams

TABLE D.1a

POC-01 Inspection of Naphtha Stabilizer Bottoms

Period	4	9	14	17	19	22	24	26	28	29	30	31
ASTM D-86 Distillation [C]												
IBP	69	66	54	57	57	58	56	60	53	60	48	58
5, V%	111	102	92	98	103	99	97	99	98	102	76	96
10, V%	137	122	115	116	123	117	116	117	116	124	92	110
20, V%	183	162	161	149	155	147	144	147	151	161	106	130
30, V%	237	204	204	185	192	179	180	180	191	202	116	149
40, V%	270	243	243	219	225	214	211	212	224	238	126	172
50, V%	293	273	271	248	255	242	243	243	251	269	138	191
60, V%	310	294	296	276	276	266	264	266	274	289	151	211
70, V%	326	311	314	296	294	285	283	283	293	307	164	227
80, V%	342	328	331	320	313	301	299	302	310	324	181	241
90, V%	362	347	352	347	332	319	319	322	329	344	207	268
95, V%	379	359	364	368	347	332	333	336	343	360	232	318
EP	391	380	382	373	367	356	352	357	366	379	301	373
Distribution, W%												
IBP-177C	15.6	19.7	19.7	24.8	22.2	24.9	25.5	25.1				
177-288C	28.5	32.5	21.8	37.1	40.1	42.7	43.7	43.9				
288-343C	35.6	32.6	42.4	35.4	29.4	27.2	25.6	26.2				
343C+	19.9	14.6	15.6	2.0	7.9	4.3	3.6	4.2				
Loss	0.4	0.6	0.6	0.7	0.4	0.9	1.6	0.6				
Distribution, V%												
IBP-177C	18	23	23	28	26	29	29	29				
177-288C	29	34	23	37	40	43	43	43				
288-343C	34	31	41	33	28	25	25	25				
343C+	18.5	11.5	12	2	5.5	2.5	2	2.5				
Whole												
API	29.6	30.3	30.6	32.6	32.0	33.0	33.3	32.8				
Carbon, W%	86.47	87.90		87.14	86.95	86.97						
Hydrogen, W%	12.03	12.66		12.45	12.55	12.61						
Nitrogen, W%	0.0273	0.0257		0.0386	0.0394	0.0306	0.0416	0.0352				
Sulfur, W%	0.0491	0.0172		0.0121	0.0139	0.0116	0.0120	0.0126				
IBP-177C Fraction												
API	52.6	52.2	51.5		52.3			51.8				
Carbon, W%	85.79	85.46			85.74							
Hydrogen, W%	14.03	14.04			13.89							
Nitrogen, W%	0.00524	0.0078			0.0139			0.0106				
Sulfur, W%	0.00977	0.0064			0.0124			0.012				
177-288C Fraction												
API	31.9	31.3	32.9		30.6			30.3				
Carbon, W%	85.00	87.63			87.69			86.69				
Hydrogen, W%	12.04	12.62			12.37			12.34				
Nitrogen, W%	0.0289	0.034			0.0452			0.0429				
Sulfur, W%	0.0133	0.0082			0.0156			0.0136				
Aniline Point, C								34				
288-343C Fraction												
API	23.2	23.0	24.1		22.4			22.2				
Carbon, W%	87.60	88.45			87.85			86.16				
Hydrogen, W%	12.00	12.03			11.83			11.59				
Nitrogen, W%	0.0345	0.0404			0.0492			0.0416				
Sulfur, W%	0.0369	0.0098			0.0137			0.0123				
Aniline Point, C								40				
343C+ Fraction												
API	20.0	19.5	21.9		18.1			18.5				
Carbon, W%	87.97	88.58			88.30			86.09				
Hydrogen, W%	11.78	11.71			11.57			11.27				
Nitrogen, W%	0.0406	0.0494			0.0565			0.0441				
Sulfur, W%	0.0461	0.0157			0.016			0.0134				

TABLE D.1b

POC-01 Inspection of Naphtha Stabilizer Bottoms

Period	33	34	35	37	40	41	42	43	46	47	48	49
ASTM D-86 Distillation [C]												
IBP	63	58	64	58	54	53	58	56	53	53	52	59
5, V%	107	107	101	101	101	102	100	96	103	98	98	101
10, V%	128	130	118	123	121	122	121	115	121	114	116	118
20, V%	172	177	152	163	156	155	154	150	153	144	146	148
30, V%	210	224	188	205	198	192	194	191	191	176	176	181
40, V%	236	253	221	237	232	226	227	226	224	209	204	211
50, V%	255	274	248	266	261	247	253	252	256	241	231	237
60, V%	269	290	269	283	279	272	272	274	279	263	256	259
70, V%	281	305	288	302	296	288	289	291	299	284	276	279
80, V%	293	319	304	320	310	304	305	307	316	303	293	298
90, V%	307	338	321	343	327	323	323	327	337	325	313	316
95, V%	320	356	334	372	342	336	338	342	352	344	329	333
EP	336	364	366	382	357	359	356	357	372	356	351	350
Distribution, W%												
IBP-177C								23.06				25.72
177-288C								41.48				46.47
288-343C								29.43				23.06
343C+								5.57				4.17
Loss								0.46				0.58
Distribution, V%												
IBP-177C	22	20	26	25				26				29
177-288C	56	29	44	38				41				46
288-343C	20	43	27	27				28				22
343C+	1.5	7.5	3	10				4				3
Whole												
API								32.5				32.5
Carbon, W%								86.37				86.3
Hydrogen, W%								12.36				12.29
Nitrogen, W%								0.0581				0.0836
Sulfur, W%								0.0345				0.0323
IBP-177C Fraction												
API								51.1				
Carbon, W%								85.12				
Hydrogen, W%								13.38				
Nitrogen, W%								0.0239				
Sulfur, W%												
177-288C Fraction												
API								30.2				
Carbon, W%								86.49				
Hydrogen, W%								12.19				
Nitrogen, W%								0.076				
Sulfur, W%								0.0394				
Aniline Point, C												
288-343C Fraction												
API								23.2				
Carbon, W%								86.86				
Hydrogen, W%								11.66				
Nitrogen, W%								0.0569				
Sulfur, W%								0.0221				
Aniline Point, C												
343C+ Fraction												
API								19.8				
Carbon, W%								86.95				
Hydrogen, W%								11.48				
Nitrogen, W%								0.0871				
Sulfur, W%								0.0252				

TABLE D.1c

POC-01 Inspection of Naphtha Stabilizer Bottoms

Period	53	54	55	56	57	Trailer Front	Trailer Front	Trailer Middle	Trailer Rear
ASTM D-86 Distillation [C]									
IBP	58	55	59	59	53	63	63	57	58
5, V%	93	90	97	95	103	100	99	91	97
10, V%	115	111	121	117	121	118	118	107	113
20, V%	1257	142	151	147	152	154	149	136	142
30, V%	181	177	181	179	184	188	188	173	173
40, V%	214	212	214	213	216	221	220	206	203
50, V%	244	242	241	240	243	248	248	234	231
60, V%	269	269	265	266	268	270	268	262	255
70, V%	288	289	285	285	284	288	286	282	276
80, V%	307	309	306	303	300	306	303	301	294
90, V%	328	330	328	328	317	327	322	324	315
95, V%	348	352	350	341	333	343	337	341	330
EP	354	368	353	360	354	354	353	356	354
Distribution, W%									
IBP-177C				24.09	24.46	23.81	23.9	27.8	27.2
177-288C				44.32	44.46	43.79	44.7	43.2	45.9
288-343C				25.57	25.6	26.25	27	23.7	22.4
343C+				5.22	4.91	5.69	4.3	5	4.1
Loss				0.8	0.57	0.46	0.1	0.3	0.4
Distribution, V%									
IBP-177C	28	30	28	28	28	26	27	31	31
177-288C	42	39	45	44	45	44	44	42	46
288-343C	24	25	21	25	24	25	26	23	20
343C+	6	6	6	3	3	5	3	4	3
Whole									
API				30.1	30.3	32.9	32.7	32.7	31.8
Carbon, W%				86.46	86.7	86.31	86.95	86.96	86.95
Hydrogen, W%				11.78	11.87	12.36	12.43	12.38	12.08
Nitrogen, W%				0.1635	0.1419	0.0549	0.0582	0.0717	0.1225
Sulfur, W%				0.0483	0.0324	0.0264	0.0329	0.033	0.507
IBP-177C Fraction									
API					50	51			
Carbon, W%					85.29	85.23			
Hydrogen, W%					13.55	13.78			
Nitrogen, W%					0.0626	0.0175			
Sulfur, W%					0.031	0.144			
177-288C Fraction									
API					28	30.4			
Carbon, W%					84.24	84.09			
Hydrogen, W%					11.15	11.67			
Nitrogen, W%					0.01778	0.0651			
Sulfur, W%					0.0519	0.0264			
Aniline Point, C									
288-343C Fraction									
API					19.8	23.5			
Carbon, W%					87.99	86.01			
Hydrogen, W%					11.12	11.52			
Nitrogen, W%					0.1722	0.0612			
Sulfur, W%					0.0374	0.0168			
Aniline Point, C									
343C+ Fraction									
API					15.3	19.8			
Carbon, W%					88.75	88.14			
Hydrogen, W%					10.49	11.52			
Nitrogen, W%					0.1978	0.706			
Sulfur, W%					0.0444	0.289			

TABLE D.2

POC-01 INSPECTION OF O-13 BOTTOMS (O-46)

Period	4	9	14	17	19	22	24	26	43	49	50	56	57
PFL, W%	62.30	57.75	56.36	68.04	64.46	74.22	73.00	73.12	71.32	62.66	54.13	58.99	69.24
PFS, W%	37.70	42.25	43.64	31.96	35.54	25.78	27.00	26.88	28.68	37.34	45.87	41.01	30.76
Pressure Filter Liquids													
Boiling Point Distillation (ASTM D-1160 Distillation) [C]													
API	10.7	9.7	11.1	11.5	10.0	11.6	10.7	10.2	6.9		4.6	0.2	1.0
IBP	306	307	303	269	282	280	285	287	311	280	284	307	278
5, V%	343	342	338	315	324	318	327	331	332	331	288	338	328
10, V%	360	361	353	341	340	339	343	348	349	346	332	354	343
20, V%	383	377	379	372	367	362	364	369	367	372	344	376	368
30, V%	403	394	394	391	383	379	379	385	384	387	372	394	387
40, V%	421	407	414	410	399	394	396	399	402	404	390	411	404
50, V%	437	427	431	427	413	410	410	413	423	425	406	433	428
60, V%	455	443	451	446	432	422	432	436	444	444	427	477	457
70, V%	474	460	476	457	454	446	456	456	489	483	451	509	501
80, V%	520	505	524	497	485	466	494	481	524	524	487	524	524
90, V%	524	524		524	524	524	524	524			524		
95, V%	(81%)	(83%)	(80%)	(85%)	(87%)	(88%)	(85%)	(87%)	(77%)	(78%)	(77%)	(73%)	(74%)
EP													
Weight Distribution, W% O-46													
IBP-343C	3.51	3.63	3.58	7.15	6.83	8.56	6.83	6.73	6.49	5.56	4.22	3.68	6.36
343-454C	31.68	32.45	28.86	33.95	38.03	42.77	41.38	40.65	35.38	31.64	25.92	27.68	31.96
454-524C	13.65	11.18	11.47	15.12	11.15	11.70	12.48	14.04	10.40	10.14	8.95	8.74	10.70
524C+	13.28	9.80	11.99	11.61	10.25	10.66	12.10	11.41	18.77	14.96	14.73	18.62	19.91
loss	0.19	0.69	0.46	0.20	0.19	0.53	0.22	0.29	0.28	0.36	0.31	0.27	0.32
Toluene Insol. W% O-46	0.14	0.13			0.17	0.20	0.22	0.24			1.42		2.76
Elemental Analysis, W% PFL													
Carbon	88.87	89.48		88.94	88.30	89.07	88.39	88.47	87.65	88.54	88.24	88.86	89.40
Hydrogen	10.47	10.09		10.35	10.36	10.30	10.33	10.17	9.75	9.62	9.35	8.41	8.58
Nitrogen	0.200	0.061		0.180	0.230	0.190	0.260	0.230	0.370	0.390	0.500	0.590	0.570
Sulfur	0.011	0.011		0.073	0.073	0.041	0.052	0.070	0.142	0.243	0.264	0.255	0.250
Pressure Filter Solids													
Quinoline Insol., W% O-46	20.23	25.49	23.64	15.31	16.78	13.73	13.56	13.02	19.32	14.76	15.95	14.92	16.09
QI Ash, W% O-46	13.09	19.00	16.78	11.21	12.18	9.80	9.57	9.08	13.68	10.12	10.27	10.50	11.61
ASTM Ash, W% O-46	13.27	18.95		11.85	12.14	9.78	9.44	9.52	13.48	10.21	10.42	10.65	11.77
S in Ash, W% Ash	2.03	1.67											
Mo in Ash, ppm Ash	213	252			163			84					186
TGA Data, W% O-46													
IBP-524C	15.18	13.65	16.20	12.72	14.79	10.03	11.25	11.28	5.17	12.31	17.51	16.64	10.04
524C+	22.52	28.60	27.44	19.24	20.75	15.75	15.75	15.60	23.51	25.03	28.36	24.37	20.72
Ash	12.62	19.32	16.77	12.07	12.64	10.03	9.65	9.60	14.12	10.46	10.50	10.62	11.85
Elemental Analysis, W% PFS													
Carbon	56.58	46.44		54.01	57.42	52.58	56.18	55.56	43.39	62.46	66.48	64.22	53.41
Hydrogen	5.53	4.09		5.20	5.55	4.91	5.57	5.28	3.41	5.61	5.69	5.25	4.29
Nitrogen	0.39	0.23		0.28	0.32	0.27	0.34	0.34	0.42	0.65	0.65	0.63	0.51
Sulfur	2.81	2.81		2.98	1.29	2.62	2.58	2.54	2.33	2.20	2.04	2.14	2.98
Whole Sample, W% O-46													
IBP-524C	64.21	61.60	60.57	69.15	69.00	73.59	72.14	72.99	57.72	60.01	56.91	57.01	59.38
524C+ (solid-free)	15.56	12.91	15.78	15.54	14.22	12.68	14.29	13.99	22.96	25.24	27.14	28.07	24.53
Unreacted Coal	7.14	6.48	6.87	4.10	4.60	3.93	4.00	3.94	5.65	4.64	5.68	4.42	4.48
Ash	13.09	19.00	16.78	11.21	12.18	9.80	9.57	9.08	13.68	10.12	10.27	10.50	11.61
Coal Conversion, W%	95.50	93.50	96.60	94.70	96.50		97.90	96.30	97.80	95.90	94.60	95.40	95.40
Elemental Analysis, W% O-46													
Carbon	76.70	71.30		77.78	77.33	79.66	79.69	79.62	74.96	78.80	78.26	78.76	78.33
Hydrogen	8.61	7.56		8.70	8.65	8.91	9.04	8.86	7.93	8.12	7.67	7.11	7.26
Nitrogen	0.272	0.132		0.212	0.262	0.211	0.282	0.260	0.384	0.487	0.569	0.606	0.552
Sulfur	1.066	1.194		1.002	0.506	0.706	0.735	0.734	0.770	0.974	1.079	1.028	1.090

TABLE D.3

POC-01 INSPECTION OF O-13 BOTTOMS (O-46)

Period No.	1	2	3	6	7	8	12	21	29	40	42	46	48	54
PFL, W%	85.78	82.13	70.17	75.93	71.33	51.34	64.87	69.92	71.62	61.67	59.63	69.88	64.73	65.99
PFS, W%	14.22	17.87	29.83	24.07	28.67	48.66	35.13	30.08	28.38	38.33	40.37	30.12	35.27	34.01
Pressure Filter Liquid														
API	13.70	12.70	11.80	16.30	12.80	10.70								
TGA Analysis														
IBP, [C]				290	294	285	296	276	283	282	279	285	286	286
IBP-524C, W% O-46	74.85		53.62			40.91	48.47	58.19	57.25	47.60	48.57	53.10	50.42	47.84
524C+, W% O-46	10.93		16.55			10.43	16.40	11.73	14.37	14.07	11.06	16.78	14.31	18.15
Pressure Filter Solid														
TGA Analysis														
IBP-524C, W% O-46	6.30		10.01	10.15	9.86	21.01	12.93	12.52	11.19	15.28	16.38	9.38	13.37	11.15
524C+, W% O-46	7.92		19.82	13.92	18.81	27.65	22.20	17.56	17.19	23.05	23.99	20.74	21.90	22.86
Ash, W% O-46			10.77	5.82	10.69	16.57	10.22	10.74	10.34	14.36	11.38	13.38	10.99	14.30
Quinoline Insol, W% O-46	6.64	10.48	17.26	12.38	16.68	22.80	17.38	14.91	14.03	19.66	15.46	18.02	15.16	19.16
QI Ash, W% O-46	3.54	6.04	10.19	5.57	10.22	16.57	9.90	10.45	10.04	13.94	10.63	13.29	10.80	14.00
S in QI Ash, W% Ash	1.61	1.82	1.48	1.81	1.67	1.86								
Whole Sample, W% O-46														
IBP-524C	81.15		63.63			61.91	61.40	70.71	68.44	62.89	64.95	62.48	63.79	58.98
524C+ (solid-free)	12.21		19.11			15.29	21.22	14.38	17.53	17.45	19.59	19.50	21.05	21.85
Unreacted Coal	3.10	4.44	7.07	6.81	6.46	6.23	7.48	4.46	3.99	5.72	4.83	4.73	4.36	5.16
Ash	3.54	6.04	10.19	5.57	10.22	16.57	9.90	10.45	10.04	13.94	10.63	13.29	10.80	14.00

TABLE D.4a

POC-01 Inspection of Atmospheric Still Bottoms

Period	4	9	12	14	17	19	21	22	24	26	29
API	20.1	19.3	18.8	19.9	17.3	18.2	19.3	18.8	18.4	18.2	17.4
ASTM D-1160 Distillation [C]											
IBP	182	232	236	199	222	205	245	240	248	248	262
5, V%	333	328	306	298	289	288	304	303	301	297	301
10, V%	346	343	330	326	318	319	314	314	314	310	316
20, V%	359	359	346	346	336	341	327	324	330	328	331
30, V%	368	368	357	361	351	347	343	334	340	340	341
40, V%	376	376	368	373	364	356	350	347	346	349	348
50, V%	383	384	378	382	376	368	359	355	354	361	359
60, V%	391	392	387	390	388	376	368	364	367	371	372
70, V%	399	401	397	399	402	386	380	377	375	379	381
80, V%	404	404	411	411	412	398	389	390	385	392	391
90, V%			427	428	431	412	404	408	399	413	409
95, V%			443	438	444	428	417	421	414	429	426
EP			480	463	471	449	441	442	446	457	450
Distribution, W%											
IBP-288C	0.50	0.30		3.42	3.89	4.44					
288-343C	7.60	8.80	17.30	15.29	19.24	20.00	31.38	37.37	31.57	33.02	31.37
343-454C	91.70	90.70	80.47	79.79	75.18	74.07	67.88	60.72	67.80	64.55	66.53
454C+			2.02	1.07	1.48	1.28	0.74	1.49	0.63	1.80	1.89
Loss	0.20	0.20	0.21	0.43	0.21	0.21	0.22	0.42		0.63	0.21
Distribution, V%											
IBP-288C	1	0.5	3	3	4	4	2	2	2	2.5	2
288-343C	8	10	18	15	20	20	32	38	32	34	33
343-454C	91	89.5	80	81	74		67	60	67	65	65
454C+			1.5		1.5			1.5	1		1.5
Whole											
API	20.1	19.3			17.3	18.2		18.8	18.4	17.4	
Carbon, W%	87.72	88.67			88.3	88.26		87.93	88.2	88.21	
Hydrogen, W%	12.00	11.68			11.32	11.49		11.49	11.54	11.58	
Nitrogen, W%	0.0466	0.0393			0.0664	0.0899		0.046	0.0607	0.0567	
Sulfur, W%	0.0494	0.0151			0.0236	0.0238		0.0142	0.0132	0.0153	
IBP-288C Fraction											
API						27.2					
Carbon, W%						87.23					
Hydrogen, W%						12.01					
Nitrogen, W%						0.0455					
Sulfur, W%						0.013					
Aniline Point [C]											
288-343C Fraction											
API	23	22.5				20.4				20.6	
Carbon, W%	87.17	88.38				88.19				87.45	
Hydrogen, W%	11.97	12.00				11.47				11.63	
Nitrogen, W%	0.0385	0.0392				0.0469				0.0392	
Sulfur, W%	0.0325	0.0144				0.0124				0.0123	
Aniline Point [C]						40.9				40.6	
343-454C Fraction											
API	19.7	19.0				18.3				17.9	
Carbon, W%	87.54	88.87				88.36				87.95	
Hydrogen, W%	11.92	11.75				11.44				11.25	
Nitrogen, W%	0.0502					0.0661				0.0583	
Sulfur, W%	0.0466					0.0146				0.0132	

TABLE D.4b

POC-01 Inspection of Atmospheric Still Bottoms

Period	40	42	43	46	48	49	50	54	56	57
API	21.3	19.8	19.6	18.5	18	17.7		14.4	13.5	14.3
ASTM D-1160 Distillation [C]										
IBP	262	248	249	263	264	274		254	272	281
5, V%	299	297	303	307	303	304		305	297	301
10, V%	321	311	318	324	314	319		323	318	311
20, V%	335	327	335	343	331	334		339	334	328
30, V%	344	343	346	351	339	343		352	345	334
40, V%	352	347	353	3795	348	348		362	354	347
50, V%	363	358	359	372	358	357		374	364	358
60, V%	373	371	370	381	373	371		381	374	368
70, V%	384	381	379	392	380	382		389	384	379
80, V%	394	392	393	404	394	393		401	397	391
90, V%	413	407	413	427	412	412		417	414	412
95, V%	429	424	428	438	429	429		438	429	430
EP	458	450	463	470	458	458		458	468	463
Distribution, W%										
IBP-288C										
288-343C	27.97	31.02	27.32	19.94	32.63	29.37		22.68	25.86	32.23
343-454C	70.19	67.7	70.86	78.26	66	69.37		74.95	71.1	65.71
454C+	1.51	1.07	1.39	1.7	1.37	1.26		1.65	1.83	1.44
Loss	0.33	0.21	0.43	0.1				0.72	0.61	0.62
Distribution, V%										
IBP-288C		3	2		2					
288-343C	29	32	28	20	33	30		23	27	34
343-454C	70	67	71	79	66	69		76	72	65
454C+				1	1	1		1		
Whole										
API			19.6							
Carbon, W%			87.85				88.47		89.19	89.54
Hydrogen, W%			11.64				11.04		10.21	10.52
Nitrogen, W%			0.0857			0.1217	0.1337		0.2413	0.2351
Sulfur, W%			0.0308			0.049	0.0512		0.0892	0.0722
IBP-288C Fraction										
API										
Carbon, W%										
Hydrogen, W%										
Nitrogen, W%										
Sulfur, W%										
Aniline Point [C]										
288-343C Fraction			21.9							
API			87.22							88.58
Carbon, W%			11.59							10.96
Hydrogen, W%			0.0608							0.1656
Nitrogen, W%			0.0198							0.386
Sulfur, W%										
Aniline Point [C]										
343-454C Fraction										
API			19.3							
Carbon, W%			88.06							89.18
Hydrogen, W%			11.4							10.21
Nitrogen, W%			0.0891							0.2485
Sulfur, W%			0.0222							0.0661

TABLE D.5

INSPECTION OF VACUUM STILL OVERHEADS

Period	4	9	17	19	22	24	26	43	49	50	56	57
API	14.9	9.1	16.9	15.4	15.8	15.2	15	16.4	14.3		9.6	9.8
ASTM D-1160 Distillation [F]												
IBP	277	274		253			271	271		274		279
5, V%	323	314		307			308	323		310		318
10, V%	341	328		328			331	337		334		334
20, V%	358	348		346			351	349		347		353
30, V%	374	366		366			367	368		366		373
40, V%	386	377		377			379	380		376		381
50, V%	396	388		389			388	389		385		391
60, V%	410	400		399			397	398		396		400
70, V%	423	412		411			409	411		404		412
80, V%	439	431		424			424	429		417		427
90, V%	463	474		441			443	443		434		443
95, V%	511	524		454			454	456		454		454
EP	524			483			484	487		481		494
	(97%)	(94%)										
Distribution, W%												
IBP-343	11.07	15.51		16.51			15.63	13.96		15.26		13.09
343-482C	73.42	66.30		82.04			82.30	83.33		82.89		85.11
482C+	14.79	16.60		1.25			1.86	2.40		1.44		1.70
Loss	0.72	1.59		0.20			0.21	0.31		0.41		0.10
Distribution, V%												
IBP-343	11	17		17			16	14		16		14
343-482C	75	70		82			82	84		83		85
482C+	14	13		1			1.5	2		1		1
Elemental Analysis, W%												
Carbon	87.92	85.77	88.12	88.42	88.46	88.43	88.51	88.39	88.59	88.75	89.48	89.48
Hydrogen	10.98	10.18	11.28	11.01	11.24	11.05	11.09	11.09	10.74	10.60	9.73	9.74
Nitrogen	0.0697	0.0772	0.0664	0.0899	0.0745	0.0894	0.0754	0.107	0.1447	0.1885	0.268	0.2908
Sulfur	0.0313	0.0411	0.0236	0.0238	0.0224	0.0185	0.0164	0.0375	0.0541	0.087	0.1367	0.108

TABLE D.6

Inspection of ROSE Bottoms (O-63)

Period No.	14	15	17	19	21	22	24	26	43	46	47	48	49	50	54	56	57
Elemental Analysis, W%																	
Carbon	65.84		72.30	56.70		51.77	45.89	47.05	50.00				56.33	59.87		61.54	62.80
Hydrogen	6.49		7.54	4.76		3.80	2.95	3.22	3.21				3.73	3.90		4.07	4.30
Nitrogen	0.34		0.31	0.43		0.49	0.52	0.54	0.66				0.87	0.98		0.97	1.04
Sulfur				2.81			3.43	3.30	3.81							2.68	2.47
TGA Analysis. W%																	
IBP-524C	32.10	8.97	37.78	17.46	13.90	11.55	7.83	9.14	6.06	18.11	17.67	6.07	5.49	6.82	7.52	9.30	9.69
524C+	67.90	91.03	62.22	82.54	86.10	88.45	92.18	90.86	93.94	81.89	82.33	93.93	94.51	93.18	92.48	90.70	90.31
Ash	24.99	46.81	17.61	34.77	39.65	39.84	45.84	44.38	40.54	22.92	33.38	35.28	33.50	29.56	29.50	28.74	27.41
ASTM Ash, W%	24.36	45.97	17.36	33.91		40.18	46.12	44.55	39.70				33.12	29.20		28.90	27.12
S in Ash, W%								1.56	0.94								1.75
Mo in Ash, ppm	214		224	39		148	131	156	213				63	60		326	116
Solubility, W%																	
Toluene Insol.	37.61	71.68	26.67	50.41	60.11	60.05	69.48	68.99	66.75	42.27	46.75	55.85	58.13	56.12	54.19	51.04	54.92
Quinoline Insol.	37.19	64.45	25.19	47.60	54.81	56.22	65.00	62.94	56.53	36.39	45.84	48.36	46.79	43.39	42.26	39.54	38.20
QI Ash	25.65	44.76	17.27	34.83	38.82	39.58	45.75	44.24	40.07	23.04	33.50	34.45	33.15	28.22	28.30	28.41	26.59

TABLE D.7

POC-01 INSPECTION OF DEASPHALTED OIL (O-65)

Period	17	19	21	24	26	43	49	56	57
PFL, W%		90.61			99.42	99.47			99.33
PFS, W%		9.39			0.58	0.53			0.67
	Whole	PFL	Whole	Whole	PFL	PFL	Whole	Whole	PFL
Boiling Point Distillation (ASTM D-1160 Distillation) [C]									
API	-3.8	6.9	-6.1	5.1	4	6.8	3.2	0	0.2
IBP	384	393	379	388	381	254	384	383	390
5, V%	412	417	413	412	421	368	411	402	410
10, V%	421	432	432	422	434	413	423	417	426
20, V%	457	450	451	447	455	436	444	440	447
30, V%	461	456	458	459	460	457	467	461	472
40, V%	472	467	479	483	472	476	488	478	488
50, V%	498	485	499	500	489	495	509	501	508
60, V%	524	504	524	516	512	517	524	517	522
70, V%		520		524	524	524		524	524
80, V%		524							
90, V%									
95, V%									
@524C	(60%)	(71%)	(57%)	(66%)	(68%)	(67%)	(60%)	(66%)	(61%)
Weight Distribution, W% PFL									
IBP-454C	12.46	21.04	21.68	22.1	17.72	26.3	25.22	27.51	23.18
454-524C	40.16	48.43	30.09	40.74	46.93	36.56	33.11	35.69	36.97
524C+	46.84	30.14	47.7	36.58	34.96	36.56	41.1	36.25	39.39
loss	0.54	0.39	0.53	0.58	0.39	0.58	0.57	0.55	0.46
Toluene Insol. W% PFL					0.9	0.48		0.6	0.7
Elemental Analysis, W% PFL									
Carbon	78.25	88.73		88.4		88.08	88.29	88.95	89.47
Hydrogen	8.86	9.95		9.58		9.53	9.2	8.35	8.46
Nitrogen	0.23	0.21		0.31		0.26		0.48	0.46
Sulfur		0.1		0.139		0.112	0.181	0.342	0.31
Pressure Filter Solids	Whole	PFS	Whole	Whole	PFS		Whole	Whole	PFS
Quinoline Insol., W%	16.39	63.23	14.89	2.14			1.98	0.92	
QI Ash, W% PFS	10.99	39.21	8.5	0.87			0.82	0.33	
ASTM Ash, W% PFS		38.8							
S in Ash, W% Ash									
TGA Data, W% PFS									
IBP-524C		25.97			7.41				
524C+		74.03			92.59				
Ash		39.9			43.54				
Elemental Analysis, W% PFS									
Carbon		51.15							51.62
Hydrogen		4.49							3.47
Nitrogen		0.35							0.45
Sulfur		5.68							0.31

TABLE D.8a

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period	01T	02T	03T	04T	05T	06T	07T	08T
Period Start Date	10/29/93	10/30/93	10/31/93	11/01/93	11/02/93	11/07/93	11/08/93	11/09/93
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	93.19	93.19	92.60	92.40	91.97	95.18	90.37	87.95
CH4	5.45	5.45	5.75	5.98	6.25	3.20	7.63	9.54
C2H4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	0.70	0.70	0.77	0.79	0.83	0.56	1.11	1.41
C3H6	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
C3H8	0.19	0.19	0.19	0.17	0.19	0.16	0.31	0.42
C4H8	0.00	0.00	0.00	0.03	0.03	0.00	0.00	0.00
N-C4H10	0.05	0.05	0.04	0.03	0.03	0.03	0.05	0.08
I-C4H10	0.02	0.02	0.01	0.01	0.02	0.01	0.02	0.02
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.04	0.04	0.00	0.04	0.04	0.00	0.00	0.00
I-C5H12	0.00	0.00	0.00	0.01	0.09	0.00	0.00	0.01
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.01	0.01	0.02	0.02	0.02	0.01	0.00	0.02
N-Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.01	0.04	0.00	0.00	0.00
CO	0.00	0.00	0.03	0.09	0.08	0.02	0.01	0.03
CO2	0.00	0.00	0.02	0.02	0.01	0.02	0.01	0.01
H2S	0.35	0.35	0.56	0.38	0.39	0.81	0.49	0.50
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	58.75	58.75	35.82	60.58	44.27	73.41	40.41	34.26
CH4	10.84	10.84	15.34	11.79	15.03	6.79	15.70	17.45
C2H4	0.01	0.01	0.02	0.00	0.04	0.00	0.01	0.05
C2H6	6.51	6.51	10.88	6.21	9.69	3.49	10.56	11.76
C3H6	0.03	0.03	0.14	0.10	0.15	0.01	0.09	0.25
C3H8	5.30	5.30	9.39	5.11	8.22	2.67	9.29	10.85
C4H8	0.00	0.00	0.11	0.08	0.11	0.01	0.10	0.18
N-C4H10	2.86	2.86	6.10	2.37	3.79	0.77	4.59	5.90
I-C4H10	0.86	0.86	1.36	0.83	1.30	0.47	1.32	1.34
C5H10	0.00	0.00	0.00	0.09	0.00	0.00	0.00	0.05
N-C5H12	0.48	0.48	1.14	0.61	1.08	0.22	1.14	1.42
I-C5H12	0.49	0.49	0.92	0.50	0.82	0.19	0.90	1.13
Methylcyclopentanes	0.08	0.08	0.12	0.10	0.11	0.03	0.14	0.18
Cyclohexane	0.33	0.33	0.45	0.40	0.44	0.19	0.55	0.53
N-Hexane	0.21	0.21	0.31	0.22	0.30	0.11	0.36	0.47
2-3-Methyl Pentane	0.10	0.10	0.13	0.09	0.13	0.05	0.15	0.24
Other C6's and C7's	0.00	0.00	0.10	0.08	0.14	0.05	0.10	0.12
CO	0.00	0.00	0.13	0.13	0.14	0.00	0.13	1.95
CO2	0.08	0.08	0.12	0.02	0.10	0.08	0.05	0.10
H2S	13.09	13.09	17.43	10.70	14.14	11.45	14.39	11.76

TABLE D.8b

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period	09T	10T	11T	12T	13T	14T	15T	16T
Period Start Date	11/10/93	11/11/93	12/04/93	12/05/93	12/06/93	12/07/93	12/08/93	12/09/93
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	87.63	87.75	95.01	92.20	90.38	91.16	91.16	90.96
CH4	9.69	9.63	3.18	5.91	7.36	6.11	6.11	7.01
C2H4	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	1.46	1.46	0.54	0.97	1.21	1.25	1.25	1.12
C3H6	0.01	0.01	0.00	0.00	0.01	0.01	0.01	0.00
C3H8	0.44	0.44	0.17	0.28	0.37	0.52	0.52	0.34
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.09	0.08	0.04	0.05	0.07	0.15	0.15	0.07
I-C4H10	0.03	0.02	0.02	0.02	0.02	0.03	0.03	0.02
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.03	0.03	0.03	0.00	0.03	0.03	0.03	0.04
I-C5H12	0.02	0.03	0.00	0.00	0.00	0.02	0.02	0.00
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.00	0.01	0.00	0.00	0.00	0.11	0.11	0.01
N-Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.04	0.02	0.09	0.06	0.06	0.04	0.04	0.05
CO2	0.01	0.01	0.05	0.02	0.02	0.01	0.01	0.02
H2S	0.56	0.51	0.86	0.50	0.47	0.54	0.54	0.36
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	38.27	46.90	67.41	51.29	37.79	23.00	23.00	27.50
CH4	16.81	16.16	6.08	12.54	14.67	14.82	14.82	18.16
C2H4	0.06	0.05	0.01	0.00	0.06	0.00	0.00	0.08
C2H6	10.80	9.44	3.58	8.77	11.30	21.33	21.33	14.80
C3H6	0.23	0.19	0.04	0.10	0.27	0.28	0.28	0.34
C3H8	10.13	8.55	3.12	7.35	10.76	14.42	14.42	13.30
C4H8	0.18	0.14	0.05	0.12	0.22	0.22	0.22	0.23
N-C4H10	5.68	4.66	1.23	2.92	5.26	5.97	5.97	5.88
I-C4H10	1.43	1.06	0.75	0.88	1.34	1.45	1.45	1.32
C5H10	0.04	0.06	0.00	0.00	0.06	0.05	0.05	0.05
N-C5H12	1.38	1.18	0.57	0.71	1.19	1.04	1.04	1.23
I-C5H12	1.08	0.86	0.35	0.54	0.94	0.87	0.87	0.95
Methylcyclopentanes	0.20	0.19	0.08	0.09	0.16	0.11	0.11	0.20
Cyclohexane	0.63	0.60	0.42	0.38	0.52	0.36	0.35	0.62
N-Hexane	0.46	0.41	0.19	0.22	0.37	0.28	0.28	0.44
2-3-Methyl Pentane	0.21	0.20	0.07	0.09	0.20	0.12	0.12	0.21
Other C6's and C7's	0.14	0.14	0.13	0.09	0.11	0.08	0.08	0.17
CO	0.14	0.15	0.12	0.15	0.15	0.15	0.16	0.19
CO2	0.10	0.07	0.29	0.24	0.17	0.15	0.16	0.19
H2S	12.00	8.99	15.51	13.54	14.47	15.32	15.32	14.13

TABLE D.8c

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period Period Start Date	17T 12/10/93	18T 12/11/93	19T 12/12/93	20T 12/13/93	21T 12/14/93	22T 12/15/93	23T 12/16/93	24T 12/17/93
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	88.40	89.72	90.40	91.26	92.24	90.09	89.86	89.13
CH4	8.94	7.90	7.11	6.20	5.81	7.66	8.01	8.66
C2H4	0.02	0.01	0.01	0.01	0.00	0.00	0.01	0.01
C2H6	1.49	1.38	1.36	1.27	1.03	1.22	1.22	1.20
C3H6	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.01
C3H8	0.46	0.41	0.45	0.47	0.32	0.37	0.35	0.36
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.09	0.07	0.09	0.10	0.06	0.07	0.06	0.07
I-C4H10	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.01
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.03	0.04	0.02	0.03	0.00	0.03	0.00	0.03
I-C5H12	0.01	0.00	0.00	0.02	0.00	0.00	0.00	0.00
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.02	0.00	0.01	0.00	0.01	0.02	0.00	0.01
N-Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00
CO	0.07	0.07	0.06	0.05	0.06	0.06	0.06	0.07
CO2	0.02	0.01	0.01	0.02	0.01	0.01	0.01	0.01
H2S	0.42	0.35	0.45	0.53	0.43	0.44	0.40	0.43
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	27.50	36.36	38.56	43.37	44.33	38.59	31.45	18.23
CH4	18.16	15.99	13.95	12.48	12.48	16.11	17.44	22.19
C2H4	0.08	0.03	0.09	0.00	0.00	0.00	0.02	0.08
C2H6	14.80	12.91	11.57	10.12	10.31	11.82	13.58	17.40
C3H6	0.34	0.38	0.37	0.21	0.21	0.27	0.29	0.38
C3H8	13.30	12.22	11.37	9.15	9.63	10.71	12.04	14.50
C4H8	0.23	0.24	0.22	0.18	0.23	0.19	0.21	0.23
N-C4H10	5.88	5.35	5.30	3.82	4.08	4.80	5.51	5.53
I-C4H10	1.32	1.15	1.07	0.81	0.91	0.97	1.25	1.19
C5H10	0.05	0.01	0.07	0.03	0.03	0.02	0.01	0.05
N-C5H12	1.23	0.98	1.12	0.85	1.22	1.07	1.25	1.04
I-C5H12	0.95	0.78	0.84	0.61	0.77	0.76	0.90	0.76
Methylcyclopentanes	0.20	0.14	0.21	0.16	0.17	0.20	0.23	0.18
Cyclohexane	0.62	0.43	0.62	0.53	0.59	0.62	0.68	0.57
N-Hexane	0.44	0.33	0.43	0.32	0.36	0.41	0.46	0.37
2-3-Methyl Pentane	0.21	0.14	0.21	0.12	0.13	0.15	0.17	0.16
Other C6's and C7's	0.17	0.14	0.18	0.18	0.17	0.17	0.19	0.16
CO	0.19	0.18	0.17	0.15	0.14	0.17	0.17	0.02
CO2	0.19	0.00	0.14	3.96	0.00	0.00	0.00	0.24
H2S	14.13	12.23	13.52	12.96	14.25	12.97	14.14	16.71

TABLE D.8d

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period	25T	26T	27T	28T	29T	30T	31T	32T
Period Start Date	12/18/93	12/19/93	12/20/93	12/21/93	12/22/93	12/23/93	12/24/93	12/25/93
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	87.55	88.93	87.02	86.23	85.42	84.55	85.38	84.58
CH4	9.82	8.85	10.29	11.13	11.15	12.75	8.77	9.99
C2H4	0.00	0.00	0.00	0.01	0.00	0.00	0.02	0.01
C2H6	1.34	1.26	1.42	1.31	1.79	1.55	2.03	2.19
C3H6	0.01	0.01	0.01	0.01	0.01	0.02	0.02	0.02
C3H8	0.45	0.37	0.41	0.36	0.58	0.39	1.06	0.87
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
N-C4H10	0.07	0.07	0.07	0.06	0.12	0.06	0.35	0.09
I-C4H10	0.02	0.02	0.02	0.02	0.03	0.01	0.06	0.03
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.00	0.00	0.00	0.03	0.02	0.00	0.04	0.03
I-C5H12	0.00	0.00	0.00	0.00	0.01	0.00	0.03	0.03
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.17	0.00	0.00	0.00	0.00	0.01	0.01	0.00
N-Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
CO	0.07	0.07	0.14	0.15	0.13	0.15	0.16	0.19
CO2	0.01	0.01	0.04	0.06	0.06	0.05	0.10	0.14
H2S	0.49	0.41	0.58	0.63	0.67	0.45	1.94	1.81
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	40.80	32.44	37.43	40.39	31.08	36.16	36.16	28.57
CH4	18.68	18.00	19.37	21.40	17.89	23.49	23.49	16.09
C2H4	0.14	0.07	0.08	0.07	0.00	0.05	0.05	0.05
C2H6	11.75	12.90	11.46	10.54	12.28	12.60	12.60	11.49
C3H6	0.25	0.30	0.29	0.25	0.30	0.27	0.27	0.21
C3H8	9.56	11.57	9.29	7.76	11.66	9.19	9.19	10.67
C4H8	0.18	0.29	0.19	0.19	0.21	0.14	0.14	0.12
N-C4H10	3.80	5.28	3.81	2.70	5.45	3.28	3.28	4.91
I-C4H10	0.79	1.01	0.82	0.68	1.08	0.69	0.69	1.06
C5H10	0.01	0.02	0.01	0.01	0.06	0.01	0.01	0.01
N-C5H12	0.80	1.47	0.94	0.61	1.24	0.68	0.68	1.53
I-C5H12	0.56	0.89	0.62	0.41	0.83	0.45	0.46	0.95
Methylcyclopentanes	0.15	0.21	0.21	0.12	0.19	0.12	0.12	0.26
Cyclohexane	0.41	0.62	0.65	0.41	0.65	0.40	0.40	0.81
N-Hexane	0.30	0.43	0.40	0.29	0.59	0.27	0.27	0.60
2-3-Methyl Pentane	0.12	0.16	0.14	0.08	0.21	0.08	0.08	0.26
Other C6's and C7's	0.13	0.17	0.18	0.12	0.22	0.11	0.11	0.19
CO	0.18	0.19	0.27	0.32	0.21	0.31	0.31	0.25
CO2	0.08	0.20	0.34	0.43	0.49	0.30	0.30	0.43
H2S	11.33	13.79	13.50	13.21	15.36	11.36	11.36	21.53

TABLE D.8e

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period	33T	34T	35T	36T	37T	38T	39T	40T
Period Start Date	01/03/94	01/04/94	01/05/94	01/06/94	01/14/94	01/21/94	01/29/94	01/30/94
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	92.65	91.72	92.29	62.42	77.08	95.99	95.30	91.39
CH4	3.62	6.11	5.67	13.79	8.36	2.79	3.10	6.49
C2H4	0.00	0.00	0.01	0.05	0.01	0.00	0.00	0.00
C2H6	0.82	1.13	1.09	6.89	3.88	0.42	0.54	1.07
C3H6	0.01	0.01	0.02	0.19	0.03	0.00	0.00	0.00
C3H8	0.46	0.36	0.36	5.57	2.56	0.13	0.16	0.32
C4H8	0.00	0.00	0.00	0.20	0.02	0.00	0.00	0.00
N-C4H10	0.18	0.07	0.07	2.86	0.94	0.03	0.03	0.06
I-C4H10	0.05	0.02	0.02	0.54	0.25	0.01	0.01	0.02
C5H10	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00
N-C5H12	0.03	0.01	0.00	0.39	0.12	0.01	0.00	0.02
I-C5H12	0.04	0.00	0.00	0.57	0.12	0.00	0.00	0.00
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.01	0.00	0.00	0.33	0.05	0.00	0.00	0.00
N-Hexane	0.01	0.00	0.00	0.16	0.03	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.06	0.01	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.16	0.12	0.11	0.09	0.20	0.11	0.16	0.13
CO2	0.11	0.04	0.02	0.14	0.23	0.04	0.06	0.00
H2S	1.83	0.41	0.35	5.73	6.11	0.47	0.64	0.49
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	66.75	50.78	40.09	42.98	51.67	82.00	93.23	54.51
CH4	7.51	13.37	16.21	18.20	12.77	4.76	1.80	12.92
C2H4	0.02	0.00	0.07	0.09	0.03	0.02	0.00	0.01
C2H6	5.38	10.09	12.88	10.99	8.97	2.48	1.31	8.39
C3H6	0.08	0.26	0.40	0.34	0.12	0.01	0.01	0.07
C3H8	3.68	8.59	11.29	9.20	6.70	1.63	1.04	6.90
C4H8	0.07	0.31	0.26	0.25	0.11	0.02	0.06	0.08
N-C4H10	1.01	3.37	4.61	3.98	2.03	0.49	0.68	2.64
I-C4H10	0.43	0.84	0.97	0.93	0.74	0.37	0.27	0.71
C5H10	0.00	0.00	0.02	0.05	0.00	0.00	0.00	0.00
N-C5H12	0.28	0.69	0.86	0.82	0.48	0.19	0.26	0.78
I-C5H12	0.20	0.56	0.64	0.61	0.37	0.16	0.16	0.48
Methylcyclopentanes	0.04	0.12	0.14	0.15	0.06	0.04	0.10	0.11
Cyclohexane	0.23	0.44	0.51	0.43	0.28	0.23	0.40	0.42
N-Hexane	0.12	0.25	0.31	0.31	0.18	0.10	0.17	0.25
2-3-Methyl Pentane	0.06	0.05	0.12	0.15	0.09	0.04	0.06	0.12
Other C6's and C7's	0.06	0.12	0.16	0.13	0.08	0.10	0.13	0.10
CO	0.19	0.24	0.26	0.26	0.31	0.14	0.04	0.23
CO2	0.42	0.23	0.20	0.23	0.52	0.21	0.30	0.28
H2S	13.43	9.70	10.01	9.90	14.50	7.02	0.00	11.02

TABLE D.8f

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period	41T	42T	43T	44T	45T	46T	47T	48T
Period Start Date	01/31/94	02/01/94	02/02/94	02/03/94	02/05/94	02/06/94	02/07/94	02/08/94
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	89.90	91.29	89.49	89.49	93.01	90.21	90.96	90.24
CH4	7.66	6.52	8.28	8.28	5.40	7.86	6.98	7.66
C2H4	0.01	0.00	0.01	0.01	0.00	0.01	0.01	0.02
C2H6	1.24	1.05	1.11	1.11	0.78	1.16	1.05	1.12
C3H6	0.01	0.01	0.01	0.01	0.00	0.01	0.01	0.02
C3H8	0.35	0.29	0.28	0.28	0.20	0.26	0.26	0.28
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.06	0.06	0.05	0.05	0.03	0.06	0.05	0.05
I-C4H10	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.03	0.01	0.02	0.02	0.01	0.01	0.01	0.01
I-C5H12	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.00	0.01	0.01	0.01	0.02	0.00	0.07	0.03
N-Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.17	0.17	0.19	0.19	0.14	0.11	0.14	0.14
CO2	0.05	0.05	0.06	0.06	0.03	0.01	0.04	0.04
H2S	0.51	0.52	0.49	0.49	0.37	0.29	0.41	0.38
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	38.14	41.69	34.48	34.48	48.13	33.66	18.18	16.15
CH4	16.43	14.73	19.10	19.10	14.02	17.71	18.13	18.56
C2H4	0.00	0.08	0.09	0.09	0.03	0.12	0.11	0.16
C2H6	11.56	10.13	13.48	13.48	10.41	14.45	15.69	16.25
C3H6	0.28	0.29	0.37	0.37	0.15	0.45	0.74	0.73
C3H8	9.93	8.81	11.27	11.27	7.96	12.24	14.38	15.18
C4H8	0.17	0.22	0.23	0.23	0.11	0.19	0.39	0.46
N-C4H10	4.29	4.13	5.04	5.04	2.35	4.77	6.36	6.99
I-C4H10	0.93	0.88	1.05	1.05	0.59	0.99	1.31	1.34
C5H10	0.01	0.05	0.03	0.03	0.01	0.05	0.04	0.07
N-C5H12	1.01	1.08	1.16	1.16	0.50	0.96	1.31	1.71
I-C5H12	0.72	0.75	0.81	0.81	0.33	0.68	0.94	1.07
Methylcyclopentanes	0.18	0.21	0.21	0.21	0.06	0.16	0.21	0.20
Cyclohexane	0.52	0.60	0.61	0.61	0.28	0.48	0.64	0.48
N-Hexane	0.39	0.45	0.42	0.42	0.20	0.36	0.43	0.45
2-3-Methyl Pentane	0.16	0.21	0.10	0.10	0.06	0.17	0.09	0.22
Other C6's and C7's	0.11	0.17	0.15	0.15	0.10	0.11	0.19	0.12
CO	0.29	1.60	0.32	0.32	0.29	0.26	0.25	0.24
CO2	3.06	0.39	0.55	0.55	0.48	0.26	0.46	0.38
H2S	11.80	13.53	10.52	10.52	13.96	11.91	20.14	19.24

TABLE D.8g

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period Period Start Date	49T 02/09/94	50T 02/10/94	51T 02/11/94	52T 02/12/94	53T 02/13/94	54T 02/14/94	55T 02/15/94	56T 02/16/94
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	88.99	88.35	88.35	87.87	87.87	86.68	85.64	85.67
CH4	8.28	9.27	9.27	10.10	10.10	10.67	11.55	11.70
C2H4	0.02	0.02	0.02	0.00	0.00	0.02	0.02	0.01
C2H6	1.32	1.21	1.21	1.13	1.13	1.27	1.32	1.32
C3H6	0.02	0.02	0.02	0.01	0.01	0.02	0.02	0.02
C3H8	0.36	0.27	0.27	0.21	0.21	0.25	0.26	0.26
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.06	0.05	0.05	0.03	0.03	0.05	0.05	0.05
I-C4H10	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
N-C5H12	0.03	0.01	0.01	0.00	0.00	0.00	0.01	0.01
I-C5H12	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.01	0.03	0.03	0.00	0.00	0.13	0.06	0.03
N-Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.17	0.17	0.17	0.14	0.14	0.25	0.29	0.26
CO2	0.07	0.06	0.06	0.04	0.04	0.12	0.13	0.11
H2S	0.65	0.53	0.53	0.46	0.46	0.53	0.61	0.54
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	16.35	18.68	18.68	26.52	26.52	23.31	27.93	20.18
CH4	17.12	18.51	18.51	20.79	20.79	19.87	21.45	23.13
C2H4	0.15	0.33	0.33	0.08	0.08	0.18	0.16	0.17
C2H6	15.42	17.84	17.84	13.42	13.42	13.76	12.39	14.98
C3H6	0.77	0.79	0.79	0.37	0.37	0.79	0.67	0.80
C3H8	13.80	12.60	12.60	10.77	10.77	11.49	9.27	11.36
C4H8	0.46	0.42	0.42	0.23	0.23	0.43	0.42	0.38
N-C4H10	6.25	5.38	5.38	4.56	4.55	4.75	3.50	4.05
I-C4H10	1.20	1.04	1.04	1.13	1.13	1.19	0.86	0.98
C5H10	0.05	0.08	0.08	0.01	0.01	0.06	0.06	0.02
N-C5H12	1.47	1.10	1.10	0.97	0.97	1.13	1.20	0.96
I-C5H12	0.97	0.75	0.75	0.86	0.86	0.85	0.73	0.62
Methylcyclopentanes	0.25	0.11	0.11	0.14	0.14	0.13	0.20	0.16
Cyclohexane	0.59	0.30	0.30	0.48	0.48	0.36	0.53	0.40
N-Hexane	0.54	0.39	0.39	0.29	0.29	0.31	0.40	0.31
2-3-Methyl Pentane	0.24	0.20	0.19	0.08	0.08	0.18	0.21	0.14
Other C6's and C7's	0.19	0.52	0.52	0.10	0.10	0.09	0.15	0.11
CO	0.27	0.28	0.28	0.25	0.25	0.35	0.38	0.41
CO2	0.39	0.48	0.48	0.35	0.35	0.62	0.67	0.71
H2S	23.53	20.21	20.21	18.60	18.60	20.15	18.82	20.11

TABLE D.8h

RUN 260-04 PRODUCT ANALYSES
PRELIMINARY CTSL PDU DATA

COAL: Illinois #6 from Crown II Mine (HRI-6158)
CATALYST: Akzo AO-60 1/16" (HRI-6043)

Period	57T	58T
Period Start Date	02/17/94	02/18/94
VENT GAS, VOL% (N2, O2 Free Basis)		
H2	86.29	86.29
CH4	11.26	11.26
C2H4	0.02	0.02
C2H6	1.31	1[illegible]
C3H6	0.02	0[illegible]
C3H8	0.25	[illegible]
C4H8	0.00	[illegible]
N-C4H10	0.04	[illegible]
I-C4H10	0.01	[illegible]
C5H10	0.00	[illegible]
N-C5H12	0.00	[illegible]
I-C5H12	0.00	[illegible]
Methylcyclopentanes	0.00	[illegible]
Cyclohexane	0.02	[illegible]
N-Hexane	0.00	[illegible]
2-3-Methyl Pentane	0.00	[illegible]
Other C6's and C7's	0.00	[illegible]
CO	0.23	0[illegible]
CO2	0.09	0[illegible]
H2S	0.46	0.[illegible]
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)		
H2	19.69	19.[illegible]
CH4	19.08	19.[illegible]
C2H4	0.00	0.[illegible]
C2H6	13.51	13.51
C3H6	0.86	0.86
C3H8	11.25	11.25
C4H8	0.50	0.50
N-C4H10	5.55	5.55
I-C4H10	1.12	1.12
C5H10	0.05	0.05
N-C5H12	1.30	1.30
I-C5H12	0.94	0.94
Methylcyclopentanes	0.21	0.21
Cyclohexane	0.49	0.49
N-Hexane	0.47	0.47
2-3-Methyl Pentane	0.23	0.23
Other C6's and C7's	0.17	0.17
CO	0.34	0.34
CO2	6.28	6.28
H2S	17.96	17.96

APPENDIX E

Hydrotreating Scouting Tests

Run 246-350: OPERATING SUMMARY

Operating Conditions

25-May-94

Period #		1	2	3	4	5	6	7	8	9	10	11	12
Duration	h	12	24	24	24	24	24	24	24	24	24	24	24
Cumulative Run hours	h	24	48	72	96	120	144	168	192	216	240	264	288
Condition		1T	1	1	1	1	1	1	1	1	1	1	1
Date (1993)		03/05	03/06	03/07	03/08	03/09	03/10	03/11	03/12	03/13	03/14	03/15	03/16
Avg. Bed Temperature	oF	687	694	688	688	688	692	691	690	700	709	710	710
Pressure inlet	psig	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800
Feed Rate	g/h	71.33	54.71	48.96	50.75	50.42	48.71	48.71	52.92	51.13	50.63	51.75	51.33
Make-up Hydrogen	SCF/h	1.86	2.19	2.16	2.16	2.16	2.16	2.16	2.15	1.94	1.79	1.79	1.79
LHSV	kg/l/h	1.4267	1.0942	0.9792	1.0150	1.0083	0.9742	0.9742	1.0583	1.0225	1.0125	1.0350	1.0267
Catalyst Age	bbl/lb	0.0798	0.1595	0.2393	0.3191	0.3989	0.4786	0.5584	0.6382	0.7179	0.7977	0.8775	0.9572

Mass Balance

In ------------------->	g												
Feed		856.00	1313.00	1175.00	1218.00	1210.00	1169.00	1169.00	1270.00	1227.00	1215.00	1242.00	1232.00
Make-up hydrogen		53.95	126.58	125.13	125.13	125.13	125.13	125.13	124.26	112.56	103.55	103.55	103.55
Water		95.90	100.00	169.80	153.80	208.40	185.20	185.20	201.40	223.10	213.00	208.40	222.80
Total		1005.85	1539.58	1469.93	1496.93	1543.53	1479.33	1479.33	1595.66	1562.66	1531.55	1553.95	1558.35
Out ------------------>	g												
Gas		63.60	166.41	166.67	141.67	163.34	164.94	164.95	161.00	151.67	139.44	156.19	150.06
Oil		808.00	1244.00	1110.00	1160.00	1150.00	1127.00	1127.00	1215.00	1189.00	1128.00	1172.00	1159.00
Water		93.00	124.00	182.00	187.00	222.00	203.00	203.00	217.00	244.00	224.00	229.00	239.00
Total		964.60	1534.41	1458.67	1488.67	1535.34	1494.94	1494.95	1593.00	1584.67	1491.44	1557.19	1548.06
Mass Balance	W%	95.90	99.66	99.23	99.45	99.47	101.05	101.06	99.83	101.41	97.38	100.21	99.34
Normalization Factor		1.0328	1.0121	1.0092	1.0072	1.0052	0.9889	0.9888	1.0015	0.9886	1.0335	1.0150	1.0187
(Calculated from Hydrogen Balance)													

RUN 246-350: OPERATING SUMMARY

Operating Conditions

25-May-94

Period #		13	14	15	16	17	18	19	20	21
Duration	h	24	24	24	24	24	24	24	24	24
Cumulative Run hours	h	312	336	360	384	408	432	456	480	504
Condition		2T	2	2	3T	3	3	3	3	3
Date		03/17	03/18	03/19	03/20	03/21	03/22	03/23	03/24	03/25
Avg. Bed Temperature	oF	733	733	735	676	674	676	709	711	711
Pressure inlet	psig	1800	1800	1800	1800	1800	1800	1800	1800	1800
Feed Rate	g/h	49.71	50.08	50.42	51.29	49.58	51.54	45.96	51.54	50.88
Make-up Hydrogen	SCF/h	1.79	1.79	1.79	1.79	1.79	1.79	1.79	1.79	1.78752
LHSV	kg/l/h	0.9942	1.0017	1.0083	1.0258	0.9917	1.0308	0.9192	1.0308	1.0175
Catalyst Age	bbl/lb	1.1051	1.1849	1.2647	1.3445	1.4242	1.5040	1.5838	1.6635	1.7433

Mass Balance

In ------------------->	g									
Feed		1193.00	1202.00	1210.00	1231.00	1190.00	1237.00	1103.00	1237.00	1221.00
Make-up hydrogen		103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55
Water		200.40	205.30	190.60	194.20	189.60	197.80	206.40	197.80	198.20
Total		1496.95	1510.85	1504.15	1528.75	1483.15	1538.35	1412.95	1538.35	1522.75
Out ------------------>	g									
Gas		134.36	132.42	137.79	141.78	139.26	132.41	128.71	132.46	129.68
Oil		1140.00	1131.00	1163.00	1191.00	1123.00	1180.00	1065.00	1174.00	1168.00
Water		224.00	211.00	207.00	209.00	195.00	204.00	211.00	210.00	206.00
Total		1498.36	1474.42	1507.79	1541.78	1457.26	1516.41	1404.71	1516.46	1503.68
Mass Balance	W%	100.09	97.59	100.24	100.85	98.25	98.57	99.42	98.58	98.75
Normalization Factor		1.0006	1.0163	0.9943	0.9862	1.0092	1.0052	0.9907	1.0130	1.0049
(Calculated from Hydrogen Balance)										

RUN 246-350: OPERATING SUMMARY

Operating Conditions

25-May-94

Period #		22	23	24	25	26	27	28	29	30	31	32	33	34
Duration	h	24	24	24	24	24	24	24	24	24	24	24	24	24
Cumulative Run hours	h	528	552	576	600	624	648	672	696	720	744	768	792	816
Condition		5T	5	5	5	5	6T	6	6	6	7T	7	7	7
Date		03/26	03/27	03/28	03/29	03/30	03/31	04/01	04/02	04/03	04/04	04/05	04/06	04/07
Avg. Bed Temperature	oF	711	710	710	711	711	719	719	718	710	708	708	714	714
Pressure inlet	psig	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800
Feed Rate	g/h	51.92	45.38	51.21	49.54	50.04	93.67	104.54	98.08	92.04	130.83	153.08	154.58	153.04
Make-up Hydrogen	SCF/h	1.79	1.79	1.79	1.79	1.79	3.39	3.54	3.54	3.5427	4.95292	5.32532	5.32532	5.32532
LHSV	kg/l/h	1.0383	0.9075	1.0242	0.9908	1.0008	1.8733	2.0908	1.9617	1.8408	2.6167	3.0617	3.0917	3.0608
Catalyst Age	bbl/lb	1.8231	1.9028	1.9826	2.0624	2.1422	2.2219	2.3017	2.3815	2.4612	2.5410	2.6208	2.7005	2.7803

Mass Balance

In ------------------->	g													
Feed		1246.00	1089.00	1229.00	1189.00	1201.00	2248.00	2509.00	2354.00	2209.00	3140.00	3674.00	3710.00	3673.00
Make-up hydrogen		103.55	103.55	103.55	103.55	103.55	196.56	205.24	205.24	205.24	286.93	308.51	308.51	308.51
Water		201.30	194.10	183.40	202.90	201.70	328.80	326.90	356.60	318.40	498.30	487.70	501.20	505.20
Total		1550.85	1386.65	1515.95	1495.45	1506.25	2773.36	3041.14	2915.84	2732.64	3925.23	4470.21	4519.71	4486.71
Out ------------------>	g													
Gas		126.93	128.77	127.05	123.66	135.09	237.13	265.36	271.73	269.59	361.93	389.11	377.57	414.59
Oil		1176.00	1071.00	1175.00	1146.00	1188.00	2166.00	2432.00	2258.00	2192.00	3024.00	3671.00	3556.00	3536.00
Water		208.00	198.00	188.00	207.00	213.00	359.00	361.00	380.00	343.00	536.00	534.00	555.00	551.00
Total		1510.93	1397.77	1490.05	1476.66	1536.09	2762.13	3058.36	2909.73	2804.59	3921.93	4594.11	4488.57	4501.59
Mass Balance	W%	97.43	100.80	98.29	98.74	101.98	99.60	100.57	99.79	102.63	99.92	102.77	99.31	100.33
Normalization Factor		1.0205	0.9662	1.0002	0.9907	0.9645	1.0099	1.0014	1.0073	0.9717	1.0081	0.9738	1.0196	1.0118
(Calculated from Hydrogen Balance)														

RUN 246-350: OPERATING SUMMARY

Operating Conditions

25-May-94

Period #		35	36	37	38	39	40	41	42	43	44
Duration	h	24	24	24	24	24	24	24	24	24	24
Cumulative Run hours	h	840	864	888	912	936	960	984	1008	1032	1056
Condition		8T	8	8	8	9T	9	9	10T	10	10
Date		04/08	04/09	04/10	04/11	04/12	04/13	04/14	04/15	04/16	04/17
Avg. Bed Temperature	oF	714	716	715	712	713	713	713	726	727	725
Pressure inlet	psig	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800
Feed Rate	g/h	183.50	200.33	176.42	171.58	57.50	54.46	47.54	50.17	50.75	50.17
Make-up Hydrogen	SCF/h	7.08	7.08	7.11	7.11	2.01	1.75	1.79	1.79	1.78752	1.78752
LHSV	kg/l/h	3.6700	4.0067	3.5283	3.4317	1.1500	1.0892	0.9508	1.0033	1.0150	1.0033
Catalyst Age	bbl/lb	2.8601	2.9399	3.0196	3.0994	3.1792	3.2589	3.3387	3.4185	3.4983	3.5780

Mass Balance

In ------------------->	g										
Feed		4404.00	4808.00	4234.00	4118.00	1380.00	1307.00	1141.00	1204.00	1218.00	1204.00
Make-up hydrogen		409.90	412.06	412.06	412.06	116.50	101.40	103.55	103.55	103.55	103.55
Water		632.20	668.40	630.20	678.20	166.90	153.10	157.70	214.60	182.40	168.20
Total		5446.10	5888.46	5276.26	5208.26	1663.40	1561.50	1402.25	1522.15	1503.95	1475.75
Out ------------------>	g										
Gas		490.03	504.64	501.74	515.41	178.22	128.73	136.05	132.41	127.66	124.43
Oil		4448.00	4420.00	4287.00	3819.00	1286.00	1216.00	1107.00	1126.00	1142.00	1142.00
Water		650.00	660.00	650.00	700.00	213.00	169.00	173.00	215.00	200.00	181.00
Total		5588.03	5584.64	5438.74	5034.41	1677.22	1513.73	1416.05	1473.41	1469.66	1447.43
Mass Balance	W%	102.61	94.84	103.08	96.66	100.83	96.94	100.98	96.80	97.72	98.08
Normalization Factor (Calculated from Hydrogen Balance)		0.9713	1.0618	0.9655	1.0480	1.0267	1.0371	0.9794	1.0282	1.0264	1.0141

RUN 246-350: OPERATING SUMMARY

Operating Conditions

25-May-94

Period #		45	46	47	48	49	50	51	52	53	54	55	56	57
Duration	h	24	24	24	24	24	24	24	24	24	24	24	24	24
Cumulative Run hours	h	1080	1104	1128	1152	1176	1200	1224	1248	1272	1296	1320	1344	1368
Condition		11T	11	11	11	11	12T	12	12	12	12	13T	13	13
Date		04/18	04/19	04/20	04/21	04/22	04/23	04/24	04/25	04/26	04/27	04/28	04/29	04/30
Avg. Bed Temperature	oF	711	710	716	715	717	717	715	715	715	708	724	725	727
Pressure inlet	psig	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800	1800
Feed Rate	g/h	51.92	45.38	52.17	53.46	50.42	55.33	50.58	50.96	48.88	51.13	48.92	51.50	49.58
Make-up Hydrogen	SCF/h	1.79	1.79	1.79	1.79	1.79	1.79	1.79	1.79	1.78752	1.78752	1.78752	1.78752	1.78752
LHSV	kg/l/h	1.0383	0.9075	1.0433	1.0692	1.0083	1.1067	1.0117	1.0192	0.9775	1.0225	0.9783	1.0300	0.9917
Catalyst Age	bbl/lb	3.6609	3.7438	3.8266	3.9095	3.9924	4.0698	4.1473	4.2247	4.3022	4.3796	4.4594	4.5391	4.6189

Mass Balance

In -------------------->	g													
Feed		1246.00	1089.00	1252.00	1283.00	1210.00	1328.00	1214.00	1223.00	1173.00	1227.00	1174.00	1236.00	1190.00
Make-up hydrogen		103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55	103.55
Water		201.30	194.10	170.40	153.20	239.70	208.30	191.50	155.10	178.10	173.20	156.50	157.70	161.90
Total		1550.85	1386.65	1525.95	1539.75	1553.25	1639.85	1509.05	1481.65	1454.65	1503.75	1434.05	1497.25	1455.45
Out ------------------->	g													
Gas		126.42	128.59	130.65	131.96	130.39	142.54	134.27	125.71	138.33	132.55	135.70	127.25	131.12
Oil		1176.00	1071.00	1153.00	1156.00	1142.00	1274.00	1133.00	1165.00	1118.00	1171.00	1118.00	1177.00	1087.00
Water		208.00	198.00	182.00	152.00	205.00	218.00	236.00	164.00	183.00	186.00	168.00	170.00	170.00
Total		1510.42	1397.59	1465.65	1439.96	1477.39	1634.54	1503.27	1454.71	1439.33	1489.55	1421.70	1474.25	1388.12
Mass Balance	W%	97.39	100.79	96.05	93.52	95.12	99.68	99.62	98.18	98.95	99.06	99.14	98.46	95.37
Normalization Factor		1.0085	0.9602	1.0314	1.0551	1.0084	1.0020	1.0303	1.0073	0.9970	1.0035	1.0035	1.0076	1.0433
(Calculated from Hydrogen Balance)														

Run 246-350: OPERATING SUMMARY

Performance

Period #		1	2	3	4	5	6	7	8	9	10	11	12
Conversion of 480 oF+	*W%*	5.29	6.97	7.38	7.06	7.06	7.38	7.38	6.92	6.93	6.79	6.95	6.95
HDS	*W%*	N/A	91.37	91.81	91.14	96.33	97.35	98.90	98.04	96.42	94.86	99.48	99.48
HDN	*W%*	N/A	99.65	99.50	99.54	99.66	99.61	99.96	99.96	99.98	98.88	99.92	99.92
Targetted concentrations													
Conversion of 480 oF+	*W%*	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
S	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
N	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
H2 Consumption (Chemical)	*W%*	0.92	0.98	0.99	0.95	0.98	0.99	0.99	0.97	0.96	0.95	0.97	0.96
H2 Consumption (Chemical)	*SCF/b*	497.16	527.11	535.29	513.90	530.73	534.50	534.63	525.78	521.16	512.31	523.78	519.77
H2 Consumption (Metered)	*W%*	2.06	1.83	1.90	3.44	2.06	2.03	2.03	2.05	1.76	1.82	0.73	1.12
H2 Consumption (Metered)	*SCF/b*	1113.01	988.65	1024.72	1860.62	1114.67	1094.96	1095.00	1105.74	949.47	984.03	393.32	607.41

Kinetic Data

First Order Rate Constants	*kg/l/h*												
k Cracking of 480 oF+		0.0776	0.0790	0.0751	0.0743	0.0739	0.0747	0.0747	0.0759	0.0735	0.0712	0.0730	0.0730
k HDS			2.6813	2.4507	2.4605	3.3325	3.5381	4.3937	4.1638	3.4057	3.0043	5.3290	5.3321
k HDN			6.1901	5.1916	5.4637	5.7191	5.3973	7.5316	8.3383	8.7395	4.5506	7.2399	7.2430
ROT HDS			733.01	733.45	733.14	710.35	709.57	692.84	696.19	720.67	739.64	697.83	697.79
ROT HDN			683.52	694.16	689.31	685.01	693.98	662.11	652.54	657.21	728.06	683.86	683.82

RUN 246-350: OPERATING SUMMARY

Performance

Period #		13	14	15	16	17	18	19	20	21
Conversion of 480 oF+	*W%*	7.23	7.22	6.54	6.69	6.86	6.10	6.33	5.85	5.74
HDS	*W%*	N/A	96.81	94.86	N/A	93.95	93.87	N/A	98.93	95.68
HDN	*W%*	N/A	99.92	99.96	N/A	98.10	97.75	N/A	99.65	99.64
Targetted concentrations										
Conversion of 480 oF+	*W%*	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
S	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
N	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
H2 Consumption (Chemica	*W%*	0.57	0.55	0.56	0.35	0.33	0.68	0.88	0.94	0.93
H2 Consumption (Chemica	*SCF/b*	305.97	297.68	301.47	187.02	180.72	368.92	473.95	506.54	505.02
H2 Consumption (Metered)	*W%*	2.12	2.30	1.93	1.58	1.87	2.23	2.69	2.23	2.44
H2 Consumption (Metered)	*SCF/b*	1146.08	1242.38	1045.82	855.03	1010.13	1207.74	1456.23	1207.88	1318.19

Kinetic Data

First Order Rate Constants	*kg/l/h*									
k Conversion of 480 oF+		0.0746	0.0751	0.0682	0.0710	0.0704	0.0648	0.0601	0.0622	0.0601
k HDS			3.4507	2.9927		2.7814	2.8778		4.6817	3.1960
k HDN			7.1553	7.9517		3.9279	3.9104		5.8178	5.7318
ROT HDS			754.39	767.90		708.41	708.56		708.74	737.38
ROT HDN			707.48	699.15		706.06	709.14		706.22	707.57

RUN 246-350: OPERATING SUMMARY

Performance

Period #		22	23	24	25	26	27	28	29	30	31	32	33	34
Conversion of 480 oF+	W%	6.92	8.17	7.59	6.36	6.45	3.20	3.44	3.87	4.08	4.05	3.83	3.41	3.73
HDS	W%	N/A	94.65	95.74	96.77	98.78	N/A	98.65	98.93	94.63	N/A	94.28	95.64	96.04
HDN	W%	N/A	99.47	99.43	99.43	99.40	N/A	99.55	99.65	97.57	N/A	96.15	99.64	96.64
Targetted concentrations														
Conversion of 480 oF+	W%	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
S	ppm	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
N	ppm	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
H2 Consumption (Chemical)	W%	0.94	0.57	0.56	0.32	0.64	0.48	0.52	0.54	0.56	0.49	0.47	0.44	0.45
H2 Consumption (Chemical)	SCF/b	508.68	307.56	301.65	170.53	344.60	260.64	279.75	289.99	301.61	265.44	253.07	239.05	241.64
H2 Consumption (Metered)	W%	2.50	3.00	2.79	2.91	2.38	1.12	0.83	0.86	1.00	0.77	0.75	1.40	0.78
H2 Consumption (Metered)	SCF/b	1349.71	1622.06	1509.76	1572.58	1289.13	606.71	449.39	464.98	540.79	417.41	403.09	756.81	422.94

Kinetic Data

First Order Rate Constants	kg/l/h													
k Conversion of 480 oF+		0.0745	0.0774	0.0809	0.0652	0.0667	0.0608	0.0731	0.0775	0.0767	0.1081	0.1022	0.0907	0.0995
k HDS			2.6580	3.2319	3.4003	4.4134		8.9999	8.9094	5.3843		7.4882	8.1946	8.4461
k HDN			4.7603	5.3008	5.1194	5.1179		11.3110	11.0714	6.8400		8.5209	14.7158	8.8752
ROT HDS			750.91	736.05	732.57	713.39		670.19	669.73	698.02		672.07	671.79	669.80
ROT HDN			724.17	714.51	718.14	718.62		656.37	657.04	691.44		669.65	630.33	672.18

RUN 246-350: OPERATING SUMMARY

Performance

Period #		35	36	37	38	39	40	41	42	43	44
Conversion of 480 oF+	*W%*	1.64	2.14	1.99	2.56	6.19	4.01	7.07	4.84	4.76	4.81
HDS	*W%*	N/A	88.70	89.14	90.21	N/A	96.99	87.30	N/A	99.04	96.57
HDN	*W%*	N/A	93.41	93.58	93.47	N/A	99.04	98.37	N/A	99.70	99.70
Targetted concentrations											
Conversion of 480 oF+	*W%*	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
S	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
N	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
H2 Consumption (Chemical)	*W%*	0.94	0.53	0.55	0.33	0.65	0.64	0.40	0.96	0.93	0.93
H2 Consumption (Chemical)	*SCF/b*	509.03	285.57	298.98	175.74	352.94	347.54	213.61	518.98	502.12	503.13
H2 Consumption (Metered)	*W%*	0.77	0.77	0.45	0.40	0.26	1.84	2.32	2.17	2.51	2.78
H2 Consumption (Metered)	*SCF/b*	417.86	414.62	243.68	216.54	140.82	992.35	1255.47	1175.67	1356.01	1503.82

Kinetic Data

First Order Rate Constants	*kg/l/h*										
k Conversion of 480 oF+		0.0608	0.0866	0.0708	0.0890	0.0734	0.0446	0.0698	0.0497	0.0496	0.0494
k HDS			8.7351	7.8343	7.9758		3.8163	1.9620		4.7125	3.3850
k HDN			10.8953	9.6898	9.3650		5.0568	3.9134		5.8931	5.8132
ROT HDS			669.39	675.83	671.75		726.15	778.46		724.18	747.96
ROT HDN			652.75	662.20	662.49		722.22	748.18		720.80	720.53

RUN 246-350: OPERATING SUMMARY

Performance

Period #		45	46	47	48	49	50	51	52	53	54	55	56	57
Conversion of 480 oF+	*W%*	2.99	3.76	3.19	3.11	3.50	1.69	1.65	1.86	2.20	1.43	5.55	5.17	15.19
HDS	*W%*	N/A	90.38	92.31	92.75	95.59	N/A	88.53	90.03	93.27	94.63	N/A	99.34	99.19
HDN	*W%*	N/A	95.75	96.38	96.41	96.93	N/A	99.49	97.77	98.03	98.42	N/A	99.75	99.75
Targetted concentrations														
Target LHSV		1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
Conversion of 480 oF+	*W%*	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
S	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
N	*ppm*	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
H2 Consumption (Chemical)	*W%*	-0.12	-0.07	-0.10	0.02	0.02	0.96	0.91	0.66	0.97	0.53	0.51	0.48	0.51
H2 Consumption (Chemical)	*SCF/b*	-65.93	-40.01	-54.47	10.75	9.37	565.65	535.35	386.79	572.21	313.89	291.97	272.28	290.32
H2 Consumption (Metered)	*W%*	2.27	3.04	1.94	1.73	2.09	1.56	1.56	2.38	2.75	1.95	1.77	2.10	1.98
H2 Consumption (Metered)	*SCF/b*	1181.21	1583.81	1008.94	899.24	1088.47	871.08	868.63	1326.82	1532.85	1083.76	955.26	1137.51	1070.43

Kinetic Data

First Order Rate Constants	*kg/l/h*													
k Conversion of 480 oF+		0.0316	0.0348	0.0338	0.0338	0.0360	0.0188	0.0169	0.0191	0.0218	0.0148	0.0584	0.0543	0.1685
k HDS			2.1244	2.6769	2.8063	3.1473		2.1911	2.3493	2.6376	2.9905		5.1650	4.7736
k HDN			2.8668	3.4628	3.5565	3.5140		5.3472	3.8782	3.8389	4.2419		6.1792	5.9560
ROT HDS			745.59	733.76	729.13	722.94		762.53	756.36	747.79	730.23		715.39	723.66
ROT HDN			707.25	695.89	692.54	695.97		701.64	730.82	732.35	715.33		714.88	720.66

Run 246-350: OPERATING SUMMARY

Product Yields

Period #		1	2	3	4	5	6	7	8	9	10	11	12
H2S	W%	0.10	0.09	0.09	0.09	0.10	0.10	0.10	0.10	0.10	0.09	0.10	0.10
NH3	W%	0.31	0.31	0.31	0.31	0.31	0.31	0.31	0.31	0.31	0.31	0.31	0.31
H2O/CO/CO2	W%	0.39	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37
C1	W%		0.02	0.11		0.07	0.11	0.11	0.02			0.00	
C2	W%	0.03	0.06	0.07	0.05	0.06	0.07	0.07	0.06	0.06	0.05	0.06	0.06
C3	W%	0.04	0.07	0.08	0.06	0.08	0.08	0.08	0.07	0.07	0.06	0.07	0.07
C4	W%		0.20	0.38	0.23	0.31	0.38	0.38	0.23	0.25	0.22	0.25	0.24
i-C5	W%	0.02	0.03	0.03	0.02	0.03	0.03	0.03	0.03	0.03	0.02	0.03	0.03
n-C5	W%	0.05	0.09	0.10	0.08	0.09	0.10	0.10	0.09	0.08	0.08	0.09	0.08
C6-C7	W%	0.04	0.07	0.08	0.06	0.08	0.08	0.08	0.07	0.07	0.06	0.07	0.07
C1-C3	W%	0.07	0.16	0.26	0.12	0.21	0.25	0.25	0.15	0.13	0.11	0.13	0.12
C4-180	W%	1.83	2.08	2.47	2.43	2.35	2.37	2.46	2.19	2.18	2.13	2.20	2.17
180-350 oF	W%	1.66	1.53	1.62	1.92	1.62	1.53	1.62	1.63	1.63	1.63	1.63	1.63
350-480 oF	W%	22.71	22.53	22.21	22.35	22.16	22.31	22.21	22.32	22.32	22.36	22.32	22.33
480-650 oF	W%	28.76	29.25	28.12	28.01	28.18	27.46	28.12	28.26	28.26	28.30	28.25	28.27
650 oF+	W%	39.68	38.93	38.80	40.10	39.74	40.04	38.80	38.99	38.99	39.05	38.98	39.01
C4+ Gravity	API	49.92	50.27	52.87	50.06	50.98	51.61	52.87		52.20	51.96	52.25	0.77
Total		98.46	97.21	96.96	97.31	97.03	96.95	96.95	97.15	97.13	97.22	97.13	97.16
Selectivity to Products													
C1-C3	%	0.08	0.17	0.29	0.12	0.23	0.27	0.28	0.16	0.14	0.12	0.15	0.13
C4-180 oF	%	1.97	2.24	2.69	2.61	2.53	2.56	2.68	2.39	2.38	2.32	2.39	2.36
180-350 oF	%	24.41	24.24	24.18	24.03	23.92	24.14	24.18	24.28	24.29	24.31	24.29	24.30
350-480 oF	%	30.91	31.46	30.61	30.12	30.42	29.71	30.62	30.75	30.76	30.78	30.75	30.76
480-650 oF+	%	42.64	41.88	42.23	43.11	42.90	43.32	42.24	42.42	42.44	42.47	42.43	42.44

RUN 246-350: OPERATING SUMMARY

Product Yields

Period #		13	14	15	16	17	18	19	20	21
H2S	W%	0.10	0.10	0.09	0.10	0.09	0.09	0.10	0.10	0.10
NH3	W%	0.31	0.31	0.31	0.31	0.30	0.30	0.31	0.31	0.31
H2O/CO/CO2	W%	0.39	0.37	0.37	0.39	0.37	0.37	0.39	0.37	0.37
C1	W%									
C2	W%	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
C3	W%	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06
C4	W%	0.24	0.22	0.23	0.22	0.26	0.17	0.34	0.17	0.18
i-C5	W%	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
n-C5	W%	0.07	0.07	0.07	0.08	0.08	0.07	0.08	0.07	0.07
C6-C7	W%	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06
C1-C3	W%	0.11	0.11	0.11	0.12	0.12	0.10	0.11	0.10	0.10
C4-180	W%	2.52	2.49	1.84	1.84	1.87	2.44	2.63	2.45	2.07
180-350 oF	W%	2.01	2.01	1.34	1.34	1.33	2.01	2.01	2.02	1.63
350-480 oF	W%	21.99	21.99	22.08	22.04	22.00	21.38	21.33	21.44	21.73
480-650 oF	W%	27.25	27.25	27.71	27.67	27.62	26.85	26.78	26.92	27.59
650 oF+	W%	39.59	39.59	40.04	39.98	39.91	41.33	41.23	41.44	40.86
C4+ Gravity	API	51.18	51.18	50.35	50.26	50.50	49.36	49.84		49.52
Total		96.93	96.88	96.84	96.71	96.55	97.09	97.07	97.35	97.34
Selectivity to Products										
C1-C3	%	0.12	0.12	0.12	0.13	0.13	0.11	0.12	0.11	0.11
C4-180 oF	%	2.75	2.72	2.00	2.00	2.05	2.65	2.85	2.65	2.24
180-350 oF	%	24.05	24.06	24.05	24.05	24.04	23.22	23.17	23.22	23.53
350-480 oF	%	29.80	29.81	30.20	30.19	30.18	29.15	29.09	29.15	29.88
480-650 oF+	%	43.28	43.30	43.63	43.63	43.61	44.87	44.77	44.87	44.24

RUN 246-350: OPERATING SUMMARY

Product Yields

Period #		22	23	24	25	26	27	28	29	30	31	32	33	34
H2S	W%	0.10	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.09	0.10	0.09	0.10	0.10
NH3	W%	0.31	0.31	0.31	0.31	0.31	0.31	0.31	0.31	0.30	0.31	0.30	0.31	0.30
H2O/CO/CO2	W%	0.39	0.37	0.37	0.37	0.37	0.39	0.37	0.37	0.37	0.39	0.37	0.37	0.37
C1	W%					0.11				0.07	0.08	0.01		
C2	W%	0.05	0.03	0.04	0.02	0.03	0.01	0.02	0.01	0.06	0.06	0.06	0.01	0.01
C3	W%	0.05	0.06	0.05	0.04	0.04	0.03	0.03	0.03	0.08	0.08	0.07	0.03	0.03
C4	W%	0.15	0.42	0.22	0.20	0.22								
i-C5	W%	0.02	0.02	0.02	0.02	0.05		0.03	0.03	0.03	0.03	0.03		0.03
n-C5	W%	0.07	0.10	0.08	0.09	0.09	0.09	0.08	0.35	0.09	0.09	0.09	0.05	0.09
C6-C7	W%	0.05	0.16	0.11	0.03	0.12	0.07	0.07	0.07	0.08	0.08	0.07	0.03	0.07
C1-C3	W%	0.10	0.09	0.09	0.06	0.18	0.05	0.06	0.05	0.21	0.22	0.14	0.04	0.05
C4-180	W%	2.41	2.78	2.53	2.12	2.38	1.66	1.69	1.95	1.86	1.88	1.78	1.59	1.69
180-350 oF	W%	2.02	2.00	2.01	1.72	1.72	1.46	1.46	1.45	1.45	1.46	1.46	1.47	1.46
350-480 oF	W%	22.44	22.14	22.28	21.49	21.47	21.12	21.06	20.97	20.92	21.55	21.60	21.70	21.62
480-650 oF	W%	27.16	26.80	26.97	26.83	26.81	27.05	26.98	26.86	26.80	26.60	26.66	26.78	26.69
650 oF+	W%	40.45	39.91	40.16	41.06	41.02	41.75	41.64	41.45	41.36	42.72	42.81	43.00	42.86
C4+ Gravity	API	49.20	50.29	49.59	49.58	50.11	50.70	51.20		52.11	48.33	48.01	0.79	47.65
Total		97.51	96.59	96.92	96.66	96.84	98.31	98.08	97.91	97.60	98.31	98.39	98.63	98.63
Selectivity to Products														
C1-C3	%	0.11	0.10	0.09	0.06	0.20	0.05	0.06	0.05	0.23	0.24	0.15	0.05	0.05
C4-180 oF	%	2.60	3.03	2.75	2.31	2.59	1.81	1.85	2.14	2.04	2.02	1.92	1.71	1.82
180-350 oF	%	24.24	24.14	24.21	23.47	23.37	23.05	23.04	22.97	22.95	23.18	23.23	23.30	23.27
350-480 oF	%	29.34	29.22	29.30	29.31	29.18	29.53	29.51	29.43	29.40	28.61	28.67	28.76	28.73
480-650 oF+	%	43.70	43.51	43.64	44.85	44.66	45.56	45.54	45.41	45.38	45.95	46.04	46.18	46.13

RUN 246-350: OPERATING SUMMARY

Product Yields

Period #		35	36	37	38	39	40	41	42	43	44
H2S	W%	0.10	0.09	0.09	0.09	0.10	0.10	0.09	0.10	0.10	0.10
NH3	W%	0.31	0.29	0.29	0.29	0.31	0.31	0.30	0.31	0.31	0.31
H2O/CO/CO2	W%	0.39	0.37	0.37	0.37	0.39	0.37	0.37	0.39	0.37	0.37
C1	W%					0.02		0.07	0.02		
C2	W%	0.01	0.01		0.01	0.04	0.03	0.03	0.03	0.03	0.04
C3	W%	0.02	0.02	0.02	0.02	0.04	0.03	0.06	0.04	0.04	0.04
C4	W%					0.17		0.32	0.16	0.13	0.13
i-C5	W%					0.03		0.02		0.02	0.02
n-C5	W%	0.03		0.03	0.03	0.09	0.02	0.08	0.05	0.04	0.06
C6-C7	W%	0.04	0.11	0.09	0.09	0.15	0.06	0.10		0.06	0.05
C1-C3	W%	0.03	0.03	0.02	0.04	0.09	0.05	0.17	0.09	0.07	0.08
C4-180	W%	1.08	1.12	1.12	1.13	2.26	1.58	2.59	1.74	1.76	1.79
180-350 oF	W%	0.98	0.98	0.98	0.97	1.72	1.45	1.90	1.44	1.44	1.44
350-480 oF	W%	21.19	21.08	21.12	20.99	21.53	20.94	21.38	21.25	21.27	21.26
480-650 oF	W%	27.76	27.62	27.67	27.50	26.88	26.82	26.70	27.41	27.43	27.41
650 oF+	W%	43.26	43.05	43.11	42.86	41.14	41.39	40.86	41.44	41.48	41.46
C4+ Gravity	API	47.53	44.57	44.55	44.59	45.05	47.92	45.07		46.04	46.06
Total		99.01	98.50	98.65	98.10	97.01	97.40	96.46	97.26	97.33	97.31
Selectivity to Products											
C1-C3	%	0.04	0.03	0.02	0.04	0.10	0.06	0.18	0.10	0.08	0.08
C4-180 oF	%	1.16	1.20	1.20	1.22	2.46	1.74	2.82	1.90	1.92	1.95
180-350 oF	%	22.70	22.69	22.70	22.69	23.42	23.06	23.32	23.12	23.12	23.11
350-480 oF	%	29.75	29.73	29.74	29.73	29.25	29.55	29.12	29.81	29.81	29.80
480-650 oF+	%	46.35	46.34	46.34	46.32	44.77	45.59	44.56	45.08	45.08	45.07

RUN 246-350: OPERATING SUMMARY

Product Yields

Period #		45	46	47	48	49	50	51	52	53	54	55	56	57
H2S	W%	0.03	0.03	0.03	0.03	0.03	0.06	0.05	0.05	0.06	0.06	0.10	0.10	0.10
NH3	W%	0.02	0.02	0.02	0.02	0.02	0.12	0.12	0.12	0.12	0.12	0.31	0.31	0.31
H2O/CO/CO2	W%	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.37
C1	W%		0.05				0.19			0.04				
C2	W%	0.03	0.02	0.03	0.02	0.02	0.03	0.03	0.03	0.04	0.03	0.04	0.04	0.04
C3	W%	0.05	0.04	0.04	0.04	0.03	0.04	0.03	0.04	0.06	0.04	0.05	0.04	0.06
C4	W%	0.20	0.41	0.18	0.24	0.12		0.07	0.10	0.25	0.10	0.17		0.26
i-C5	W%	0.04	0.05	0.02	0.02	0.02	0.02			0.02		0.03		0.02
n-C5	W%	0.09	0.10	0.07	0.10	0.02	0.07		0.07	0.88	0.07	0.08		0.08
C6-C7	W%	0.11	0.12	0.12	0.15	0.19	0.18	0.07	0.03	0.09	0.09	0.03	0.03	0.06
C1-C3	W%	0.08	0.11	0.07	0.06	0.05	0.26	0.06	0.07	0.14	0.08	0.09	0.08	0.10
C4-180	W%	2.34	2.59	2.28	2.38	2.31	1.78	1.45	1.52	2.80	1.59	1.83	1.55	4.53
180-350 oF	W%	1.81	1.79	1.80	1.81	1.90	1.25	1.25	1.25	1.43	1.25	1.43	1.44	4.00
350-480 oF	W%	37.69	37.39	37.62	37.65	37.88	17.30	17.31	17.27	16.63	16.76	21.21	21.30	25.83
480-650 oF	W%	23.03	22.85	22.99	23.01	23.70	21.24	21.25	21.21	20.53	20.69	26.18	26.29	23.54
650 oF+	W%	27.89	27.67	27.83	27.85	27.12	43.25	43.27	43.18	43.62	43.96	42.05	42.22	38.31
C4+ Gravity	API	55.22	50.46	50.25	50.28	49.17	61.78	61.36		62.34	61.92	45.12	0.80	45.03
Total		96.14	95.64	95.88	96.06	95.99	97.21	96.90	96.77	97.14	96.67	96.74	96.84	96.61
Selectivity to Products														
C1-C3	%	0.09	0.12	0.08	0.07	0.05	0.31	0.08	0.09	0.17	0.09	0.10	0.09	0.11
C4-180 oF	%	2.57	2.85	2.51	2.62	2.53	2.13	1.74	1.82	3.35	1.91	2.01	1.70	4.91
180-350 oF	%	41.40	41.27	41.43	41.39	41.60	20.64	20.77	20.75	19.87	20.18	23.22	23.29	27.98
350-480 oF	%	25.30	25.22	25.32	25.30	26.03	25.34	25.50	25.47	24.52	24.90	28.66	28.75	25.50
480-650 oF+	%	30.63	30.53	30.66	30.63	29.79	51.59	51.92	51.87	52.10	52.92	46.02	46.17	41.50

APPENDIX F

Modifications

APPENDIX F

MODIFICATIONS

The first task initiated following contract award and environmental assessment was Task 3, PDU Modifications. Equipment upgrades were required to accomplish the objectives of the program and also to improve equipment reliability and to reduce operating costs. Modifications proceeded from November 1992 to late October of 1993. Each major item installed or modified is listed below.

Sub Task 1 - **Pretreatment Reactor** - Scheduled for Phase II (FY 1996)

Sub Task 2 - **Solids Separation Equipment** - Install a redesigned ROSE-SR^{SM} Unit with salvageable components from Wilsonville. This includes clearing the area and relocating the Dowtherm system and construction of a motor control center.

Sub Task 3 - **On-Line Hydrotreating** - Install an On-Line Hydrotreater Vessel in the Reactor Tower. This consisted of modifying a reactor vessel to accomodate two fixed beds and to process all light overhead hydrocarbons.

Sub Task 4 - **Interstage Separator** - Scheduled for Phase II.

Sub Task 5 - **Reactor Structure Modifications** - The existing Reactor Structure was too small to accomodate three reactor vessels, a hot separator and catalyst systems and in addition was enclosed with asbestos based siding. Steel support beams were added, the reactor vessels and supporting equipment was added and new siding was installed. This was a major component of the modifications.

Sub Task 6 - **Coal Handling System** - Coal silos to accomodate pulverized and dried coal prepared at another site and to deliver coal to the PDU and to receive coal from bulk truck delivery was installed.

Sub Task 7 - **Electric Power Back-up** - To minimize the chance of power failures and unit shutdowns a hook-up to a separate power supply grid with automatic switching capability was initiated.

Sub Task 8 - **Automation and Control** - The instrumentation and Computer Control System was upgraded to accomodate the additional equipment and to automate field operations. This included adding more capacity to the Micro-vax II by conversion to a Vax 3400 and adding 2 operator terminals.

Sub Task 9 - **Rebuild existing Norwalk hydrogen compressers.** - This rebuilding was necessary to regain original design capacity, to minimize leakage and maximize on stream operation.

Sub Task 10 - **New Catalyst Addition and Withdrawal Valves** - The obsolete Rockwell lubricated plug valves were replaced with the proven Valvetron metal to metal seat ball valves.

Sub Task 11 - **Safety, Ventilation and Environmental Upgrades** - Items considered as improvements not essential to personnel safety are scheduled for Phase II.

Sub Task 12 - **High Pressure Sample System** - An on-line sampling system was installed on the first stage ebullating pump line to collect a representative sample of the back mixed first stage reactor.

Sub Task 13 - **Product Fractionation Upgrades** - These changes are proposed to produce a product spectrum approaching that of a commercial facility. Scheduled for Phase II.

Sub Task 14 - **Additional Spare Ebullating Pump** - A spare ebullating pump was fabricated using a casing, stator and pump parts obtained from Wilsonville.

Other - Additional necessary modifications performed prior to POC Run 1 were the installation of new preheater burners and controls, repair of the U. S. Filter and installation of a new flare.

APPENDIX G

Water Quality: GC/MS Semi Volatile Organics

(A Report Prepared by CORE Laboratories)

Core Laboratories

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By ____

WESTERN ATLAS

CORE LABORATORIES
A N A L Y T I C A L   R E P O R T

Job Number: 940554
Prepared For:

HYDROCARBON RESEARCH, INC.
DR. S. HILDEBRANDT
P.O. BOX 6047
LAWRENCEVILLE, NJ 08648

Date: 05/26/94

REVISED REPORT

Signature ____ Date: 5/26/94

Name: Chip Meador

Title: Regional Manager

CORE LABORATORIES
1733 NORTH PADRE ISLAND DRIVE
CORPUS CHRISTI, TX 78408

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WESTERN ATLAS

Core Laboratories

LABORATORY TESTS RESULTS
05/26/94

JOB NUMBER: 940554 CUSTOMER: HYDROCARBON RESEARCH, INC. ATTN: DR. S. HILDEBRANDT

CLIENT I.D.........: HRI 260-4-44B, 0-45
DATE SAMPLED.......: / /
TIME SAMPLED.......: :
WORK DESCRIPTION...: HRI 260-4-44B, 0-45

LABORATORY I.D...: 940554-0001
DATE RECEIVED....: 03/09/94
TIME RECEIVED....: 10:00
REMARKS..........:

TEST DESCRIPTION	FINAL RESULT	LIMITS/*DILUTION	UNITS OF MEASURE	TEST METHOD	DATE	TECHN
GC/MS Semivolatile Organics		*10		EPA SW-846 8270	03/16/94	GEF
Phenol	480	100	mg/kg	EPA SW-846 8270		
4-Chloro-3-methylphenol	<200	200	mg/kg	EPA SW-846 8270		
2-Chlorophenol	<100	100	mg/kg	EPA SW-746 8270		
2,4-Dichlorophenol	<100	100	mg/kg	EPA SW-846 8270		
2,4-Dimethylphenol	790	200	mg/kg	EPA SW-846 8270		
4,6-Dinitro-2-methylphenol	<500	500	mg/kg	EPA SW-846 8270		
2,4-Dinitrophenol	<500	500	mg/kg	EPA SW-846 8270		
2-Methylphenol	540	100	mg/kg	EPA SW-846 8270		
4-Methylphenol	780	100	mg/kg	EPA SW-846 8270		
2-Nitrophenol	<100	100	mg/kg	EPA SW-846 8270		
4-Nitrophenol	<500	500	mg/kg	EPA SW-846 8270		
Pentachlorophenol	<500	500	mg/kg	EPA SW-846 8270		
2,4,5-Trichlorophenol	<100	100	mg/kg	EPA SW-846 8270		
2,4,6-Trichlorophenol	<100	100	mg/kg	EPA SW-846 8270		
Extraction - Semivolatiles (BNA)	Completed			EPA SW-846 3580	03/16/94	RAD

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(512) 289-2673

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WESTERN ATLAS

Core Laboratories

QUALITY ASSURANCE REPORT
05/26/94

JOB NUMBER: 940554 CUSTOMER: HYDROCARBON RESEARCH, INC. ATTN: DR. S. HILDEBRANDT

BNA Matrix Spike Compounds (Soil) DATE ANALYZED: 03/16/94 TIME ANALYZED: 16:27 METHOD: EPA SW-846 8270 QC NUMBER:954957

BLANKS

TEST DESCRIPTION	ANALY SUB-TYPE	ANALYSIS I.D.	DILUTION FACTOR	ANALYZED VALUE	DETECTION LIMIT	UNITS OF MEASURE
Acenaphthene	MB	031694	1	<10	10	mg/kg
4-Chloro-3-methylphenol	MB	031694	1	<10	10	mg/kg
2-Chlorophenol	MB	031694	1	<10	10	mg/kg
1,4-Dichlorobenzene	MB	031694	1	<10	10	mg/kg
2,4-Dinitrotoluene	MB	031694	1	<10	10	mg/kg
4-Nitrophenol	MB	031694	1	<20	20	mg/kg
N-Nitrosodi-n-propylamine	MB	031694	1	<10	10	mg/kg
Pentachlorophenol	MB	031694	1	<20	20	mg/kg
Phenol	MB	031694	1	<10	10	mg/kg
Pyrene	MB	031694	1	<10	10	mg/kg
1,2,4-Trichlorobenzene	MB	031694	1	<10	10	mg/kg

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BNA Matrix Spike Compounds (Soil) DATE ANALYZED: 03/16/94 TIME ANALYZED: 16:27 METHOD: EPA SW-846 8270 QC NUMBER:954957

REFERENCE STANDARDS

TEST DESCRIPTION	ANALYSIS SUB-TYPE	ANALYSIS I. D.	DILUTION FACTOR	ANALYZED VALUE	TRUE VALUE	PERCENT RECOVERY	DETECTION LIMITS	UNITS OF MEASURE
2-Fluorophenol	RS	B342.14.26	1	220	200	110	10	mg/kg
Phenol-d6	RS	B342.14.26	1	200	200	100	10	mg/kg
Nitrobenzene-d5	RS	B342.14.26	1	100	100	100	10	mg/kg
2-Fluorobiphenyl	RS	B342.14.26	1	90	100	90	10	mg/kg
2,4,6-Tribromophenol	RS	B342.14.26	1	220	200	110	10	mg/kg
Terphenyl-d14	RS	B342.14.26	1	90	100	90	10	mg/kg
Acenaphthene	RS	B342.14.26	1	90	100	90	10	mg/kg
4-Chloro-3-methylphenol	RS	B342.14.26	1	100	100	100	10	mg/kg
2-Chlorophenol	RS	B342.14.26	1	90	100	90	10	mg/kg
1,4-Dichlorobenzene	RS	B342.14.26	1	90	100	90	10	mg/kg
2,4-Dinitrotoluene	RS	B342.14.26	1	100	100	100	10	mg/kg
4-Nitrophenol	RS	B342.14.26	1	60	100	60	20	mg/kg
N-Nitrosodi-n-propylamine	RS	B342.14.26	1	120	100	120	10	mg/kg
Pentachlorophenol	RS	B342.14.26	1	130	100	130	20	mg/kg
nol	RS	B342.14.26	1	100	100	100	10	mg/kg
ene	RS	B342.14.26	1	90	100	90	10	mg/kg
1,2,4-Trichlorobenzene	RS	B342.14.26	1	90	100	90	10	mg/kg

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BNA Matrix Spike Compounds (Soil) DATE ANALYZED: 03/16/94 TIME ANALYZED: 16:27 METHOD: EPA SW-846 8270 QC NUMBER:954957

MATRIX SPIKES

TEST DESCRIPTION	ANALYSIS SUB-TYPE	ANALYSIS I. D.	DILUTION FACTOR	ANALYZED VALUE	ORIGINAL VALUE	SPIKE ADDED	PERCENT RECOVERY	DETECTION LIMITS	UNITS OF MEASURE
2-Fluorophenol	MB	940554-00	1	230	0	200	115	10	mg/kg
	SS	940554-1	1	2000	0	2000	100	10	mg/kg
	SS	940581-1	1	1900	0	2000	95	10	mg/kg
Phenol-d6	MB	940554-00	1	240	0	200	120	10	mg/kg
	SS	940554-1	1	2000	0	2000	100	10	mg/kg
	SS	940581-1	1	2400	0	2000	120	10	mg/kg
Nitrobenzene-d5	MB	940554-00	1	116	0	100	116	10	mg/kg
	SS	940554-1	1	980	0	1000	98	10	mg/kg
	SS	940581-1	1	820	0	1000	82	10	mg/kg
2-Fluorobiphenyl	MB	940554-00	1	110	0	100	110	10	mg/kg
	SS	940554-1	1	1000	0	1000	100	10	mg/kg
	SS	940581-1	1	1000	0	1000	100	10	mg/kg
2,4,6-Tribromophenol	MB	940554-00	1	230	0	200	115	10	mg/kg
	SS	940554-1	1	2400	0	2000	120	10	mg/kg
	SS	940581-1	1	2200	0	2000	110	10	mg/kg
·phenyl-d14	MB	940554-00	1	100	0	100	100	10	mg/kg
	SS	940554-1	1	820	0	1000	82	10	mg/kg
	SS	940581-1	1	1200	0	1000	120	10	mg/kg
Acenaphthene	BS	940554-00	1	24	0	20	120	10	mg/kg
4-Chloro-3-methylphenol	BS	940554-00	1	47	0	40	118	10	mg/kg
2-Chlorophenol	BS	940554-00	1	45	0	40	112	10	mg/kg
1,4-Dichlorobenzene	BS	940554-00	1	22	0	20	110	10	mg/kg
2,4-Dinitrotoluene	BS	940554-00	1	19	0	20	95	10	mg/kg
4-Nitrophenol	BS	940554-00	1	45	0	40	112	20	mg/kg
N-Nitrosodi-n-propylamine	BS	940554-00	1	16	0	20	80	10	mg/kg
Pentachlorophenol	BS	940554-00	1	26	0	40	65	20	mg/kg
Phenol	BS	940554-00	1	44	0	40	110	10	mg/kg
Pyrene	BS	940554-00	1	21	0	20	105	10	mg/kg
1,2,4-Trichlorobenzene	BS	940554-00	1	23	0	20	115	10	mg/kg

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QUALITY ASSURANCE FOOTER
05/26/94

Standard Methods for the Examination of Water and Wastewater, 17th Ed. APHA,AWWA,WPCF.
USEPA SW-846 3rd. Edition, Test Methods for the Evaluation of Solid Waste
EPA-600/4-79-020, Methods for the Analysis of Water and Wastes, March 1983
Federal Register, July 1, 1992 (40 CFR Part 136).
EPA-600/2-78-054, Field and Laboratory Methods Applicable to Overburdens and Minesoils.

Quality control acceptance criteria are method dependent.
GCMS tuning criteria meet EPA CLP Statement of Work OLM01.0.
All data reported on sample "as received" unless noted.
Sample IDs with a "-00" at the end indicate a blank spike or blank spike duplicate associated with the numbered sample.
NC = Not Calculated due to value at or below detection limit.
The data in this report are within the limits of uncertainty specified in the referenced method unless otherwise indicated.
NOTE: Data in QA report may differ from final results due to digestion and/or dilution of sample into analytical range.

The "TIME ANALYZED" in the QA Report refers to the start time of the analytical batch which may not reflect the actual time of each analysis. The "DATE ANALYZED" is the actual date of analysis.

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QC SAMPLE IDENTIFICATION

1. BLANKS	Method or Method / Type	MB or MB / type^
	Reagent	RB
	Initial Calibration	ICB
	Continuing Calibration	CCB
	Storage	SB
2. STANDARDS	Laboratory Control	LCS
	Reference	RS
	Initial Calibration	ICV
	Continuing Calibration	CCV
3. SPIKES	Matrix	MS
	Blank	BS
	Surrogate	SS
	Post Digestion Spike	PDS
	Matrix Spike Duplicate	MSD
4. DUPLICATES	Matrix	MD
	Post Digestion Duplicate	PDD

^ In the event that several different method blanks are analyzed, the blank type will be designated by the preparation method, i.e., ZHE, TCLP, 3010, 3050, etc.

Subcontracted Analysis Codes

Anaheim	*AN
Aurora	*AU
Casper	*CA
Houston	*HP
Lake Charles	*LC
Long Beach	*LB
Other Laboratories	*XX

* The asterisk in the "TECHN" column signifies that the analysis was performed by a subcontract laboratory.

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